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CHEMICAL ABSTRACTS

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1—APPARATUS

C. G. OERICK

The vapor compression system of evaporation. GUSTAV CARLSSON. *Chem. Met. Eng.* 24, 645-7(1921).—The "Autovapor" type of evaporator of Swiss manuf. depends upon compression of the vapors leaving the evaporator to a sufficient pressure and corresponding temp. so that when returned through a coil submerged in the liquor being evapd. heat transfer is set up and further vaporization obtains. Continuous evapu. takes place by the addition of mechanical energy required for compression. The development of a high speed rotary compressor was essential to this system. On a single vacuum pan the mechanical energy input is about 10% of that used in evapn. Dil. solns. give best results because of small h. p. rise with concn. Test runs in concg. NaOH from 7° to 32° Bé., evapg. 2200 lbs. of H₂O per hr., gave 37 lbs. H₂O per kw.-hr., no preheater being used. Entrainment was very small. The energy balance (circulating heat = 100%) shows 6.1% supplied by compressor, 12.4% live steam added and 10.7% removed in hot condensate available for other use. Calcs. are made comparing ordinary triple-effect, quadruple-effect and compression evaporators, the lbs. H₂O evapd. per equiv. lb. of coal being resp. 17, 25.5 and 33.6, coal being used to produce electrical energy and exhaust steam not being available. With exhaust steam available, the electrical energy input may be expressed directly in lbs. coal. In this case the compression system gives an evapn. of 95 lbs. H₂O per lb. coal while a high pressure quadruple-effect evaporator gives 63.7 lbs. H₂O per lb. coal. Large compression units have given as high as 84 lbs. H₂O per kw.-hr. (See *Z. Ver. deut. Ing.* 64, 956-8, 986-9, and *Power* 53, 238).

C. C. HERITAGE

Test code for evaporating apparatus. ANON. *Mech. Eng.* 43, I, 184-7(1921).—This code as drafted by the Power Test Committee of the A. S. M. E. is intended for equipment heated by steam such as vacuum pans and multiple-effect evaporators. The objects of the tests should be defined first since these govern choice of operating conditions and to some extent the measurements involved. The app. and instruments necessary are listed, calibration being made as per Code on this subject. The dimensions of the app. must be known together with physical condition, leakage, etc. Data to be observed and calcd. are set forth in tabular form under headings of general information, dimensions, av. pressure and temp., steam quality, total quantities, thermal efficiency, capacity, economy, heat balance, liquor analysis and physical properties of liquor. Calcs. involving excess boiling degrees, self-evapn., apparent heat transmission coeff. with corrections, and evapn. from and at 212° F. are fully explained.

C. C. HERITAGE

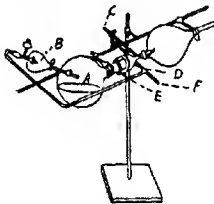
Filtration by rotary vacuum filter. F. BLOOR. *J. Soc. Chem. Ind.* 40, 82-3R(1921).—Aside from installations dealing with Na₂CO₃ and (NH₄)₂SO₄ manuf., this type of filter has found little favor (in England). Its use should be extended because the cake is accessible, being easily washed thoroughly and easily removed, and the whole operation is continuous, involving low labor costs. A filter 6.0 ft. by 3.33 ft. handles 6-8 tons Na₂CO₃ per hour for 6 d. labor charge. The superiority of this type can be demonstrated only by actual filtration tests. B. reports two examples: a light cryst. substance, cake 0.5 in. thick, drum 1.5 r. p. m., capacity 5 tons per hr. on 6.0 ft. by 3.33 ft. drum; and a very sticky sludge, 0.2 in. cake at 1 r. p. m., washing easily, capacity about 1 ton

per hr. The success of the filter depends on keeping the cloth clean which may be accomplished by "reverse flow." Such products as sludges, intermediates and dyestuffs can be filtered with this equipment at less cost than with neuteches or plate and frame filters. Sketches and description of exptl. filter are given. C. C. HERITAGE

The centrifuge in competition with the filter press. ANON. *Caliche* 1, 400(1920).—A centrifuge for intermittent continuous sepn. of liquids from solids and working on the principle of the cream separator is described. C. L. BURDICK

Centrifugal leaching apparatus. E. ALMEYDA ARROYO. *Caliche* 1, 377(1920).—An app. designed to use the effect of centrifugal force in percolation and filtration in the author's process for the leaching of caliche is described. C. L. BURDICK

Lead suction-filter sieve for laboratory use. A. COBENZL. *Nuszlösch. Chem. Ztg.* 45, 365(1921).—Pour melted Pb into a round can lid of suitable diam. until the cake is 7-8 mm. thick, cool and drill full of small holes, like the porcelain plates. This sieve is satisfactory for most purposes and there is no danger of breaking funnels that are not round when high vacuum is used. J. H. MOORE



Apparatus for filtering in moisture-free or indifferent gases. ARTHUR WOLFRAM. *Ber.* 54, 857-9(1921).—The ppt. formed by the interaction, in A in an atm. of dry air or other gas, of the two solns. one of which was held in B before mixing, may be filtered by turning the app. to a vertical position and applying suction at C. The neck of flask A rests on a porcelain filter plate, which is covered by a filter paper, and is fitted into the funnel tube D by a rubber ring at E. The ppt. may then be dried by the continued passage of the dry air or other gas, which enters at F, through the app. G. W. STRATTON

Regulating valve for gases under high pressure. FRIEDRICH MÜLLER. *Karlsruhe Z. angew. Chem.* 34, Aufsatzteil 122(1921); 1 cut.—Le Rossignol's valve (*C. A.* 2, 3170) is improved by having the upper part of the stem which carries the needle point and discharge tube screwed on the lower part which carries the valve seat, which is sprung into position so that it will adjust itself to the needle, to prevent wear on the seat and breaking of the point. A worn seat is easily replaced. The discharge tube is screwed in instead of brazed, and the packing gland between the stem and cylinder is improved. J. H. MOORE

Apparatus for determining the permeability of gas-tight materials. W. FRENZEL. *Chem. App.* 8, 57-9(1921).—Descriptions, with cuts, of the Renard-Surcouf balance, the Wurtzel app., the app. of the Zeppelin Air-ship Construction Co., of the Dresden Mechan.-Technolog. Inst., and of the Grosz-Lichterfeld Material-testing Bureau. The 1st 3 of these give too low values as compared with the results by the Zeiss interferometer and the Grosz-Lichterfeld combustion method, the relative values for the Renard-Surcouf, Wurtzel, Grosz-Lichterfeld app., and Zeiss interferometer being 9.35, 10.3, 13 and 13.5, resp. J. H. MOORE

A device for measuring the flow of gases. CARLE R. HAYWARD. *Mass. Inst. of Technology. Chem. Met. Eng.* 24, 780(1921).—A piece of 3" pipe 8' long was capped at both ends, and the caps were tapped to receive $1/4$ " inlet and outlet pipes. A tee in the outlet line connects with 1 arm of a U-tube for Hg or H_2O ; the other arm is connected with a pipe screwed into the side of the large pipe. The outlet pipe is threaded to receive a brass plug, through which a small hole is bored. A series of these plugs is kept on hand, with holes varying in size from $1/4$ " to $1/16$ ". The flow through each plug for various manometer readings was detd. with air, using a gas meter to measure the flow, or with very small vols., by displacing water in a graduated tube. By proper choice of plug and of liquid in the manometer, flows of SO_2 , H_2 , and air, in vols. varying

from 3 l. per hr. to 100 l. per min., may be measured. Manometer pressures varied from 1" of H_2O to 30" of Hg.

DONALD W. MACARDLE

A nephelometer. CH. CHÉNEVEAU AND R. AUDUBERT. *J. phys. radium* [6] 2, 19-23.—A somewhat more extended discussion of the matter treated in C. A. 14, 1621.

DONALD W. MACARDLE

Contributions from laboratory practice. O. RUDOLPH, Spandau. *Chem. Ztg.* 45, 288-9(1921); 5 cuts.—(1) *Concentration of solution by evaporation.*—This is hastened by bubbling dry air from a H_2O pump, or other source, through the boiling soln. (2) *Rapid drying apparatus for substances sensitive to high temperatures.*—The substance is placed on the shelf of a desiccator, preferably with 2 side tubulures, and the desiccator placed in a H_2O bath. $CaCl_2$ tubes with regulating cock are connected to 1 end of a coiled Ph pipe in the bath, the other end of the pipe being connected through a stopper in 1 tubulure to a short piece of rubber tubing which delivers the warm, dry air at the bottom of the desiccator. The other tubulure is connected to a manometer and suction filter flask with bits of pumice satd. with H_2SO_4 in the bottom, and a $CaCl_2$ tube between the flask and H_2O pump to catch moisture that escapes the H_2SO_4 . The bath is heated to the desired temp., the desiccator exhausted till the pressure is about 10 mm. above the vapor tension of the H_2O (or alc.) at that temp., and the cock on the $CaCl_2$ tubes set so that a slow current of air is drawn through the app. (3) *Easily constructed Bunsen burner made of glass tubing.*—The sides of a suitable tube are thickened with a pointed flame until the inside diam. is reduced to about 0.75 mm. for the gas passage. Just above this point a hole is blown equal in diam. to the inside diam. of the tube, for the air inlet.

J. H. MOORE

Thermoelements of non-precious metals for high temperature measurement. F. HOFFMANN AND A. SCHULZE. *Elektrotech. Z.* 41, 427-33(1920); *Science Abst.* 24, A, 89.—Description of expts. in which alloys of Ni, constantan, Cr, and steel were used for pyrometric measurements from 600 to 1200°.

W. H. BOYNTON

Apparatus for sodium peroxide fusions. H. J. HODSMAN. *J. Soc. Chem. Ind.* 40, 74T(1921).—The reaction vessel consists of a thin-walled steel cartridge with a tight-fitting cap. The cartridge is enclosed in an outer cylinder with a lid held in place by a bayonet catch which, when drawn up, holds the cap of the inner tube in place. The outer tube and cover are extended at both ends and bored for the insertion of a rod which facilitates opening. Combustion is effected by supporting in an inclined position over a Bunsen burner flame or by heating in a muffle.

W. H. BOYNTON

An inductance coil for use in preparing colloidal suspensions of metals by the Bredig method. A. F. C. CADENHEAD. *Can. Chem. Met.* 5, 104(1921).—A simple app. consists of soft Fe wire 2 mm. diam. and 50 cm. long bound tightly together to act as a core. On this is wound 150 ft. (45.7 m.) of Cu bell wire in 3 concentric coils, the layers being sep'd. by tape. The terminals are arranged for use of 1, 2, or 3 of the coils in parallel. The whole is taped and mounted on a wooden stand into which hindling posts are screwed. With a series connection with a Bredig arcing stand and a resistance producing at arcing 3-4 amp. on a 110-v. circuit, a continuous arc may be produced for 4-5 min. The metal dispersion is uniform in size of particles with a minimum settling out of coarse particles.

W. H. BOYNTON

A variable resistance. O. MAASS AND C. H. WRIGHT. *J. Am. Chem. Soc.* 43, 1178-9(1921).—The Pt resistance wire is sealed into an evacuated glass tube which is held vertically and is connected at its lower end with a Hg bulb. The level of the Hg in the tube contg. the wire is controlled by a suction pump connected to the Hg bulb. In the instrument used 50 cm. of wire corresponded to 1 ohm so that by reading to 0.2 mm. the accuracy is of the order of 0.002 ohm. The resistance of the Hg is taken into account by calibrating the resistance for various positions of the meniscus.

G. W. STRATTON

BOLAS, BERNARD D.: *A Handbook of Laboratory Glassblowing*. London: George Routledge & Sons. 3s. 6d. net. For review see *Chem. Trade J.* 68, 634(1921).

BÜHLER, F. A.: *Filtern und Pressen, zum Trennen von Flüssigkeiten und festen Stoffen*. 2nd Ed. revised by Ernest Jänecke. Leipzig: Otto Spamer. 172 pp. M 37. For review see *Chem. Weekblad* 18, 224(1921).

Buret. ALBERT FLEISCHAUER. Ger. 322,864, Sept. 13, 1919. To prevent the scale from becoming illegible, the measuring tube, carrying a scale which has visibly arranged graduation marks all around, is fused into a transparent tubular casing having only at the back ground a glass or light shield or similar strip, so that the scale is not only completely protected but is also at all times accessible over its entire extent and can be easily read off.

Apparatus for estimating specific gravity of a gas. G. KONIG. Brit. 159,845, Mar. 5, 1921. The d. of a gas is detd. by rotating a solid body in a chamber contg. the gas and causing the current of gas thus set up to act on a second movable solid body which operates an indicating device.

Apparatus for analyzing flue gases. HERBERT M. SHARP. Can. 211,201, May 3, 1921. The app. consists of an elec. circuit, a receptacle contg. conducting liquid, in which electrodes are submerged, a conduit delivering gas into the liquid and past one of the electrodes so that the gas bubbles emerging from the conduit press the conducting liquid away from the electrode and interrupt the circuit, and means controlled by the circuit for indicating the number of interruptions. A desired number of such receptacles is connected in series, each contg. an absorbent for a particular constituent of the gas.

2—GENERAL AND PHYSICAL CHEMISTRY

W. E. HENDERSON

Paul Jannasch. E. KNOEVENAGEL. *Chem. Ztg.* 45, 465(1921).—Obituary. E. H.

Progress in physical chemistry in 1920. W. HERZ. *Chem. Ztg.* 45, 421-3, 438-9 (1921). E. H.

Chemistry in the United States. CHARLES F. CHANDLER. *J. Ind. Eng. Chem.* 13, 391-7(1921).—An address in Rochester at the time of the recent meeting of the Am. Chem. Soc. This survey of chemistry in the U. S., made by one who has witnessed not only the beginning of the Am. Chem. Soc. but practically the beginning of chemistry in America, is of much historical interest and value. E. J. C.

History of the recovery of mercury. BERNHARD NEUMANN. *Z. angew. Chem.* 34, Aufsatzteil 161(1921). E. H.

Harmony of atomic weights and mathematics. D. DROSTE. *Pharm. Ztg.* 65, 1017-8(1920).—Reply to Schmitz's recent paper on this subject (cf. *C. A.* 14, 873 W. O. E.

Revision of the atomic weight of bismuth. A. CLASSEN AND O. NEV. *Z. anorg. allgem. Chem.* 115, 253-72(1921).—The at. wt. of Bi should be 209 instead of the older value of 208. For methods see *C. A.* 15, 1643. E. D. WILLIAMSON

Residual affinity. MATEUS D'A. ALBUQUERQUE. *Rev. chim. applicada* 4, 235-6 (1919).—A general discussion. L. E. GILSON

The fundamentals of chemical affinity. J. HEVROVSKY. *Chem. Listy* 15, 41-6 (1921).—An address. JOHN M. KRNO

Chemical affinity of the noble gases. FRANZ SKAUFY. *Z. Physik* 3, 408-11(1920).—In the light of the valence theory of Kossel (*C. A.* 14, 2883) it is probable that compounds of the noble gases with other elements can be prepd. This seems particularly true in the case of the halogens where there is one missing electron in the outer shell. One of the electrons in the complete outer shell of the noble gas might enter this space and be

held in common by the two atoms. Two stable configurations would be formed in this way. These compds. have so far escaped detection because the methods have not been careful, and because in any mixt. where the noble gas halide might be formed, conditions are also favorable to the formation of the double halide molecule. The presence of noble gases is known to have an influence upon certain reactions. S. considers this an indication of the formation of intermediate compds.

C. R. PARK

Theory of auxiliary valences and "water of crystallization." H. T. F. RHODES. *Chem. News* 122, 85-7, 97-9(1921).—Werner's theory of auxiliary valences is unsatisfactory on account of the difficulty in distinguishing between primary and secondary valences, and it is difficult to escape the conclusion that auxiliary valences have no real existence. Kohlrausch [*Proc. Roy. Soc.* 71, 338(1903)] has suggested a ring formula for H_2O of crystn., consisting of 6 H_2O mols., united through the O atoms, which are considered as quadrivalent. Examn. of a list of salts containing H_2O of crystn. shows that the most numerous class is that of salts with 6, 7, or 12 mols. of H_2O , comprising 38% of all salts listed. The ring formula for $(H_2O)_6$, taken singly or doubled, accounts for the salts with 6 and 12 H_2O . Various other speculative arrangements are proposed to account for the structure of the remaining types of hydrated cryst. salts, the formulas involving halogen or other substitution in the $(H_2O)_6$ ring, and tervalent Cl in chlorides. Numerous errors occur, both in text and in structural formulas.

M. R. SCHMIDT

A maxim covering the molecular volumes of the halogens and their compounds which holds for all states of aggregation. WILHELM BILTZ. *Z. anorg. allgem. Chem.* 115, 241-52(1921).—In extending the ideas of Fajans and Grimm (*C. A.* 15, 198) it is found that by plotting the mol. vols. of the halogens or the halides of a large no. of metals, as well as org. halogen derivs., against the mol. vol. of the corresponding K halide, a straight line will connect the points whose equation is therefore: $V_{R\text{ hal}} = aV_{K\text{ hal}} + b$. Values of a and b are tabulated. Usually F is anomalous. It is possible to det. approx. the mol. vol. of any halide once the slope of the line is established by known values for any of the series. Or relations such as $V_{MBr_2} = 1.1545 V_{MCl_2} + b$ may be established. Other homologous elements or families such as the alkali metals do not permit of any such simple treatment.

G. L. CLARK

A comparison of Tammann's and Cuy's theories concerning the periodic irregularities of the physical properties in homologous series. EUSTACE J. CUY. *Univ. Calif. Z. anorg. allgem. Chem.* 115, 273-87(1921).—The explanation by Tammann (*C. A.* 14, 2469) of the fact that the m. ps. of the fatty acids as the no. of C atoms increase advance by alternate rise and lowering with an even no. of C atoms causing a higher m. p. than the succeeding odd, is that the compds. with even no. exist in 2 enantiotropic forms with different m. ps., while those with an odd no. exist in only one form. This theory is criticized because it is based only upon the exptl. knowledge that formic acid has only one while acetic acid has 2 modifications; because such a theory merely shifts the question to why enantiotropism exists with even nos. of C atoms; and because it is by no means comprehensive enough to explain similar periodic irregularities in an even-odd system such as coord., soly., mol. rotation and change of b. p. with change in no. of C atoms. With this is contrasted the theory depending upon the disposition of elec. charges around the C atoms and the changes when increments of CH_2 or C_2H_4 resp. are made. This has been developed in *C. A.* 14, 1120.

G. L. CLARK

Periodic irregularities of the physical properties in homologous series. G. TAMMANN. *Z. anorg. allgem. Chem.* 115, 288-9(1921); cf. *C. A.* 14, 2469 and preceding abstr.—Reply to criticism of Cuy.

G. L. CLARK

The relation between crystallographic properties and the constitution question in the case of some organic compounds. A. SCHLEICHER. *J. prakt. Chem.* 100, 49-56(1919); The limited "free rotatory power" from the standpoint of the theory of symmetry. *Ibid* 57-64.—The first of these 2 papers suggests, by analogy with the supposed tetrahe-

dral arrangement of the groups about the C atom of a methane deriv., a 6-faced (either rhombohedral or scalenohedral) arrangement of the groups of an ethane deriv. The second paper discusses various compds. in the light of this idea. R. W. G. WYCKOFF

Note on the production of a contracting clot in a gel of gelatin at the iso-electric point. D. J. LLOYD. *Biochem. J.* 14, 534-5(1920); cf. *C. A.* 14, 1919.—L. has developed the theory that "gelatin gels consist of a solid framework of pure uncombined gelatin, with an interstitial fluid which consists of a soln. of gelatin salts, either acidic or basic." L.'s expts. show that gelatin gels are unstable at the iso-electric point. NaOH in small quantities has the same stabilizing influence as small quantities of HCl. Only in the presence of electrolytes is the formation of stable gels possible. B. H.

The motion of a sphere of oil through carbon dioxide and an exact determination of the coefficient of viscosity of that gas by the oil-drop method. L. J. LASSALLE. Univ. of Chicago. *Phys. Rev.* 17, 354-66(1921); cf. *C. A.* 7, 3701.—The value of ϵ being known, through work in air, to within about 0.1%, the oil-drop method as developed by Millikan has been used in CO₂ for the detn. of the viscosity of that gas with result at 23° $\eta = 1.490 \times 10^{-4}$. The value of the const. A in Millikan's equation $e_1 = e(1 + A/\alpha)^{1/2}$ is found in CO₂ to be 0.8249 against Millikan's value in air 0.864. Precisely as in Millikan's work A was found to be const. only up to $1/\alpha = 0.5$. D. MACRAE

Mobility and association of electrical carriers in flame gases. FRANZ LAUSTER. *Z. Physik* 3, 396-403(1920).—The gases from a flame are passed through a tubular electrical condenser. The mobility of the conducting bodies in the gases varies with the voltage until a certain max. value is reached. The mobility ω is calcd. by means of the following equation, $\omega = [(r_o^3 - r_i^3)/2L] \ln(r_o v/r_i V)$, where L is the length of the inner electrode of the condenser, r_o and r_i are the radii of the outer and inner cylindrical electrodes, v is the velocity of the gases, and V the max. or satn. potential. A series of measurements at various temps. and velocities of the gases shows that there is a critical temp. for each gas burned, above which ω increases with the temp. ω also increases with the velocity of flow of the gas until the velocity reaches 100 cm. per sec. where turbulent flow begins. It is noted that there are two types of carriers, one of which reaches a max. mobility before the other. One of these is probably a group which splits up at higher temps. C. R. PARK

Anhydrous yellow ferric oxide. JOHN H. YOE. *J. Phys. Chem.* 25, 196-200(1921); cf. *C. A.* 11, 3406.—Fe₂O₃ in fine particles is yellow. The usual red appearance is due to agglomeration, which can be prevented by stabilizing agents. Among these are discussed, with results: alumina, effective; CaSO₄ or BaSO₄, effective when present in great excess (94%); alumina and BaSO₄, effective when present to the extent of 10 and 84% or more, resp. Attempt was made to stabilize with alumina and later dissolve alumina in NaOH of various concns. Most of the alumina was removed but change in color to dark reddish brown indicated agglomeration. G. R. J.

A new method for the investigation of absorption of gases by solids. R. SEELIGER. *Z. Physik* 4, 189-94(1921).—S. describes a method for detg. accurately, by the change in period of a torsion pendulum, the mass of gas absorbed by a solid. If the substance is a powder it is put in the space between two light coaxial cylinders of Al, which are suspended by a Pt wire. From the fractional change in the period the fractional change in the total moment of inertia and mass can be found. F. C. HOYT

The action of heat on colloidal solutions. DEZEINE. *J. pharm. Belg.* 2, 146-148(1921).—D. reviews the theories which have been advanced to explain the coagulation of colloids by heat. A. G. DUMÉZ

Ultramicroscopic structure of soaps. W. F. DARKER, J. W. MCBAIN AND C. S. SALMON. *Proc. Roy. Soc. (London)* 98A, 395-409(1921).—The ultramicroscopic observations of Zsigmondy and Bachmann (*C. A.* 7, 1644) are confirmed, interpreted and

extended. The cinematograph was employed as an aid in elucidating the formation and disappearance of the various structures observed. Curds of Na soap consist of a felt of hydrated fibers enmeshing and in equil. with a soap sol or gel of definite concn., the soly. rising rapidly with temp. The individual fibers may be many cm. long, but they are barely of microscopic diam. K soap solns., on cooling, first develop fibers which are similar to those of the Na soaps, except that they are only a few hundredths of a mm. in length, and that they have a strong tendency to form twins. The stable condition at room temp. is, however, the formation of innumerable tiny lamellar crystals of hydrated soap. Cetylsulfonic acid ($C_{16}H_{33}SO_3H$) is similar to the K soaps, but the particles of colloidal $C_{16}H_{33}SO_3H$ are very much more prominent.

C. C. VAN VOORHIS

Contribution to the solubility problem. I. FRITZ EPHRAIM. Univ. Berne. *Ber. 54B*, 379-85 (1921).—The influence of solvation of the ions on the soly. of a salt in H_2O is discussed, and it is concluded that since it is not possible to obtain difficultly sol. ions (i. e., ions as ppts.), it is probable that the ion carries a thicker mantle of H_2O than does the mol., and in consequence non-ionized mols. are miscible with the solvent to a much smaller extent. When the mol. is capable of taking up solvent, e. g. in the form of subsidiary valence compds., it is then sol. even though undissociated. Thus, compds. containing H_2O of crystn. are usually sol. in H_2O . If a connection exists between the capacity of the solute (ion or mol.) to take up solvent and the soly., the latter must be related to the structure of the mol. Several applications of these considerations are dealt with briefly. The formation of ppts. is probably due to changes in constitution; there exists a marked difference between the insoly. of salts of the ester type, to which perhaps belong the carbonates and phosphates of the heavy metals, and the slightly sol. salts of the ion type, as represented by K_2PtCl_6 , $KClO_4$ and similar compds. It is concluded that if a salt possesses the ester form it is practically insol., while if it is of the ionizable type it is sol. to a greater or lesser degree. II. Ammine polyiodides. III. Iodides of bismuth and mercury amines. FRITZ EPHRAIM AND PAUL MOSIMANN. *Ibid* 385-96, 396-401.—A number of the conclusions reached in the preceding paper have been confirmed by the results of measurements of the stability of metal-ammine polyiodides. These results afford information as to how the I_2 , BiI_3 and HgI_2 mols. are taken up by the metal amines. The following new polyiodides, the methods of prepn. of which are given, were employed in the measurements: $[Ni(NH_3)_4]I_{3.1}$ to $6I_2$, $[Zn(NH_3)_4]I_{2.2I_2}$, $[Cd(NH_3)_4(H_2O)]I_{2.2I_2}$, $[Cu(NH_3)_4]I_{2.4I_2}$, $[Co(NH_3)_4]I_{2.3I_2}$, $[Co(NH_3)_4(CO)]I_{2.1I_2}$, *cis*- and *trans*-polyiodides of $[Co(NH_3)_4(NO_2)_2]I_{2.1I_2}$, $[Co(NH_3)_4(CI)]I_{2.2BiI_3}$, $[Co(NH_3)_4(NO_2)]I_{2.1BiI_3}$, $[Co(NH_3)_4(CO)]I_{2.1BiI_3}$, $[Ni(NH_3)_4]I_{2.1HgI_2}$, $4H_2O$, $[Ni(NH_3)_4]I_{2.1HgI_2}$, $8H_2O$, $[Ni(NH_3)_4]I_{2.1HgI_2}$, $2H_2O$, $[Ni(NH_3)_4]I_{2.1HgI_2}$, $8H_2O$, $[Zn(NH_3)_4]HgI_4$, $[Cd(NH_3)_4]HgI_4$, $4H_2O$, $[Cd(NH_3)_4]HgI_4$, $6H_2O$, $\{[Co(NH_3)_4(CO)]I\}_{2.1HgI_2}$, $[Co(NH_3)_4(NO_2)]I_{2.1HgI_2}$, $[Co(NH_3)_4]I_{2.1HgI_2}$ and $[Co(NH_3)_4]I_{2.1Fe(CN)_4}$. With ammine-pentiodides of bivalent metals the firmness with which the I mol. is held increases with the at. vol. of the metal. IV. Ammines of salts of picric acid and of *p*-dichlorobenzenesulfonic acid. FRITZ EPHRAIM. *Ibid* 402-6.—The paper contains a brief description of the methods of prepn. of the following amines of salts of picric acid: $[C_6H_3(NO_2)_3O]_2[Ni(NH_3)_4(H_2O)_2]$, $[C_6H_3(NO_2)_3O]_2[Cu(NH_3)_4(H_2O)_4]$, $[C_6H_3(NO_2)_3O]_2[Cd(NH_3)_4(H_2O)_4]$, $[C_6H_3(NO_2)_3O]_2[Zn(NH_3)_4(H_2O)_4]$, $[C_6H_3(NO_2)_3O]_2[Ag(NH_3)_2]$, and of Ni , Cu , Cd , and Zn amines of *p*-dichlorobenzenesulfonic acid.

H. JERMAIN CREIGHTON

Absorption of gases in the electric discharge tube. F. H. NEWMAN. *Proc. Phys. Soc. (London)* 33, 73-82 (1921); cf. *C. A.* 14, 2285.—In this investigation the various elements tested were deposited in a pure condition on the cathode in an elec. discharge tube, by distn. *in vacuo*. The relative absorption of Ph , Tl , As , Hg , Cd , Sh , Bi , Mg , Ca , Zn , Sn , P , S and I for both N and H under the influence of the elec. discharge was measured by comparing the amt. of gas absorbed by the element in the tube with the amt. of

H liberated by the current passing through an electrolytic cell. N was absorbed by all of the elements above mentioned except Pb, Tl and As and in comparatively large amts. by P, S, and I. H was absorbed only by P, S, and I. The absorption is attributed almost entirely to chem. action brought about by the active modifications of N and H produced under the influence of the elec. discharge at the low pressures used.

C. C. VAN VOORHIS

The oxidation of arsenious acid by nitric acid in the presence of mercuric ions. A case in which a negative catalyzer changes into a positive catalyzer. ALFONS KLEMENC AND FRIEDERICK POLLAK. Vienna. *Z. anorg. allgem. Chem.* 115, 131-40(1921).—Qual. expts. suffice to show the marked effect of Hg^{++} in inhibiting the oxidation of H_3AsO_3 by HNO_3 , but quant. measurements brought out the fact that the effect of Hg^{++} was dependent on its concn. At a concn. of 7×10^{-8} mol. per l., the oxidation is prevented entirely; between 7.7×10^{-8} and 7.7×10^{-9} the effect passes through zero; and at a concn. of 7.7×10^{-11} mol. per l. Hg^{++} acts as a strong positive catalyzer.

GEORGE W. MOREY

Catalysis. VII. Temperature coefficient of physiological processes. NIL RATAN DHAR. Muir Central College, Allahabad, India. *Proc. Roy. Acad. Sci. Amsterdam* 23, 44-9(1920); cf. *C. A.* 12, 111; 13, 3052; 14, 2286.—Physiol. processes usually occur in a heterogeneous medium. The Brownian movement of the colloidal particles, which are present in the reacting substances, renders unnecessary the assumption of a diffusion layer, which is characteristic of heterogeneous reactions, and makes physiol. reactions similar to positively catalyzed reactions occurring in a homogeneous medium. Therefore, the temp. coeffs. of physiol. processes, instead of being small (approx. 1.2), are usually greater than 2 for an increase of 10° in temp. The spontaneous destruction of certain toxins is greatly influenced by temp.; this phenomenon is extremely useful to the human body, since a toxin is very rapidly destroyed by fever. Before the destructive action of temp. begins, the formula of Arrhenius for the relation between temp. and reaction velocity applies generally to physiol. processes. VIII. NIL RATAN DHAR, A. K. DATTA AND D. N. BHATTACHARYA. *Ibid* 299-307.—Reaction between silver nitrate and ferrous ammonium sulfate: This reaction, in dil. solns., is bimol., is very rapid even at a temp. of 0° , and has a small temp. coeff. Finally an equil. is attained since Ag and $\text{Fe}(\text{NO}_3)_3$ react to form AgNO_3 and $\text{Fe}(\text{NO}_3)_2$. The reaction is accelerated by acids, including H_2CO_3 , is not influenced by H_3BO_3 and PhOH , and is retarded by MnSO_4 and KNO_3 ; in the series HNO_3 , H_2SO_4 , AcOH , citric and tartaric acids, the acceleration becomes greater as the H-ion concn. increases. While a neutral soln. of glucose does not reduce AgNO_3 at a temp. of approx. 20° , the reaction between AgNO_3 and $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$ induces the reaction between glucose and AgNO_3 . Oxidation of sodium sulfite by atmospheric oxygen: In the oxidation of Na_2SO_3 , sucrose, lactose, glucose, camphor and menthol act as negative catalysts, while naphthalene and anthracene have no action. Practically the same effect is produced by an org. acid and by its Na salt; thus "weak" acids like AcOH , propionic and cacodylic acids, and their Na salts exert a comparatively small retarding action, while comparatively "strong" acids like benzoic, oxalic and salicylic acids, and their Na salts exert far greater retarding action. The temp. coeff. of the reaction is approx. 2, and is not appreciably changed by the presence of hydroquinone, a strong negative catalyst. The sulfite ion is the active agent in the oxidation; the org. compds. act as negative catalysts by forming stable complexes with this ion and thereby decreasing its concn.; the complexes are not oxidized as readily as the ion. Catalytic activity of the undissociated acid: Study was made of the hydrolysis of Me acetate by oxalic acid in the presence of K oxalate, and by picric acid in the presence of Na picrate. The velocity coeff. decreased in the presence of oxalate; it first increased, then decreased as the concn. of the picrate became greater. These results are explained on the basis of the increases in dielectric const.

and ionizing power of the solvent which occur when salts are dissolved in the latter.

IX. Thermal and photochemical reactions. NIL RATAN DHAR. *Ibid* 308-12.—Evidence is presented that reactions which have a large temp. coeff. are sensitive to light. Hence, sensitiveness to temp. and to light radiations accompany each other. The reaction between K oxalate and I is influenced most by the indigo portion of the spectrum; the blue and violet regions exert more influence than the red region on the reactions between NH_4 oxalate and the following compds.: HgCl_2 , FeCl_3 , KMnO_4 , also the reaction between quinine bisulfate and $\text{H}_2\text{Cr}_2\text{O}_7$. Salts of U function as general positive catalysts in photochem. reactions, while salts of Mn act as general negative catalysts in both the presence and absence of light. A soln. of cupric ammonium oxalate is decomposed with the sepn. of metallic Cu and the evolution of CO_2 by tropical sunlight, but not by the light of the C arc, or of the arc with electrodes composed of ZrO_2 and ThO_2 .

X. Explanation of some abnormally large and small temperature coefficients. NIL RATAN DHAR. *Ibid* 313-8.— HIO_3 and KI in dil. soln. react very rapidly even at a temp. of 0° ; the temp. coeff., which cannot be detd. exactly, is probably equal to 1. When this reaction occurs in the presence of Na_2SO_4 or MgSO_4 , slightly less I is liberated at higher than at lower temps.; increased hydrolysis of these sulfates occurs at higher temps. and increases the number of OH ions, which react with the nascent I and regenerate iodide and iodate; in this manner the temp. effect is partly counteracted. The temp. coeff. of the reaction is not influenced by MnSO_4 , K_2SO_4 , or alum, but is increased to a value greater than 1 by the presence of $(\text{NH}_4)_2\text{SO}_4$ or ZnSO_4 , which produce more H-ions at higher temps.; these H-ions are very active in liberating I from HIO_3 and KI. The presence of OH ions, derived from the added salt, explains the small temp. coeff. in reaction between HIO_3 and KI in the presence of AcONa , KF , or Na_2HPO_4 , between CrO_3 and oxalic acid in the presence of Na_2SO_4 , MgSO_4 , or NaF , and between H_2O_2 and HI in the presence of NaHCO_3 , and also explains the abnormally large values of the temp. coeff. of the reaction between I and the OH ion with the production of the iodide and iodate ions in the presence of Na_2CO_3 and NaHCO_3 . The reactions between KI and K oxalate, KI and $\text{K}_3\text{Fe}(\text{CN})_6$, and KI and HNO_3 have measurable velocities and their temp. coeffs. are greater than 1 even in the presence of MgSO_4 or HCl . The reaction between KI, KMnO_4 , and H_2SO_4 , and that between KI and either Br or Cl are almost instantaneous even at a temp. of 0° , and have temp. coeffs. of approx. 1.

JOSEPH S. HEPBURN

The equilibrium between nitric oxide, nitrogen peroxide, and aqueous solutions of nitric acid. CHARLES L. BURDICK AND E. STANLEY FREED. *J. Am. Chem. Soc.* **43**, 318-30(1921).—The authors find that the fundamental reaction in the conversion of nitrous gases to nitric acid by absorption in aq. solns. of nitric acid is $3\text{NO}_2 + \text{H}_2\text{O} = 2\text{HNO}_3 + \text{NO}$. In the study of this reaction the equil. const. $K = (\text{P}_{\text{NO}})(\text{P}_{\text{HNO}_3})^2 / (\text{P}_{\text{NO}_2})^3 (\text{P}_{\text{H}_2\text{O}})$ was detd. for the temps. 10, 25, 50, and 75° at which temps. $\log K$ was found to be, respectively, -2.32 , -2.34 , -2.38 , and -2.39 . These consts. were obtained by combining the experimentally detd. "partial" consts. $K_1 = (\text{P}_{\text{NO}})/(\text{P}_{\text{NO}_2})^3$ and $K_2 = (\text{P}_{\text{HNO}_3})^3 (\text{P}_{\text{H}_2\text{O}})$, which vary with the acid concn. in the liquid phase. The first of these partial consts. relates to the compn. of the nitrous gases while the second relates only to the partial pressures of HNO_3 and water vapors over the soln. From the const. K and previously known free energy values, the free energy of HNO_3 gas was calcd. as $-25510 + 31T$. From the partial consts. K_1 , the vapor pressures of HNO_3 over the solns. and the free energies of HNO_3 in the solns. at the temps. 25, 50, and 75° were calculated for acids of strengths varying from 5 to 70% by wt. From the partial consts. K_2 , the authors calc. and present in convenient tabular form the % conversion of nitrogen peroxide gases (NO_2 concn. in gas varying from 0.1 to 20.0%) to HNO_3 when absorbed in aq. solns. of HNO_3 (acid concn. varying from 5 to 70%) at the temps. 10, 25, 50, and 75° .

E. S. FREED

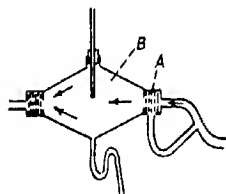
A mechanical model for the representation of thermodynamic processes and entropy in gases. RICHARD V. DALLWITZ-WEGENER. *Physik Z.* 22, 103-9(1921).—The model consists of a rolling wt. which rests on a revolving platform the rate of rotation of which may be altered by a ratchet arrangement. The wt. is held from sliding off the platform by a string which passes over a pulley at the center of rotation and finally after passing over another pulley is attached to a second wt. which rests on an inclined plane. The energy of the first wt. due to rotation is analogous to the internal energy of a gas and the work done by the second wt. in sliding on the plane is equiv. to the external work so that an analogy exists between the mechanical equations of such motions and the thermodynamic relations for a gas. The analogy is worked out fully for the usual types of processes—isosteric, adiabatic, isothermal and isobaric—and for cycles consisting of such processes.

E. D. WILLIAMSON

The temperature of the vapor arising from boiling saline solutions. GEORGE HARKER. *J. Proc. Roy. Soc. N. S. Wales* 54, 218-26(1920).—The vapor from a soln. of 220 g. of CaCl_2 in 400 g. H_2O (which boils normally at 115°) passed up the central tube of a hypsometer, having one thermometer *B* just above its upper opening and another *A* half way up. The central tube was jacketed by the vapor itself as it passed down a larger concentric tube to a condenser, and the condensed water was returned automatically to maintain the concn. of the soln. After boiling the CaCl_2 soln., *A* gradually rose to 106° and *B* to 103.8° . Analogous results were obtained by boiling with live steam. "The results prove that the vapor arising from a boiling soln. has a higher temp. than the vapor from the boiling pure solvent. Since in these expts. the vapor was cooled after it left the soln., there can be little room for doubt that it leaves the soln. at the same temp. as the boiling soln. itself. It is therefore steam in an unsatd. condition.

JEROME ALEXANDER

Rectification by adiabatic condensation without expansion. E. CHENARD. *Bull. soc. chim.* 25, 546-52(1919).—By the distn. of alc.- H_2O mixts. through this Cu app. any liquid transported in the vesicular state is removed at *A'* by metallic sieves, and a partial condensation of the vapor occurs in *B*. A series of such distns. shows that the law of correspondence between the strength of liquids and vapors in contact is almost identical with the law of fractionation by vaporization. On the distn. of 54% of an alc.- H_2O mixt. the original vapor from which contains 35% alc., through a series of 6 of the app. shown, the vapor is enriched to 64% alc., and each of the six fractions collected gives on boiling a vapor richer in alc. than the preceding fraction.



G. W. STRATTON

Distillation of aqueous solutions of formaldehyde. JOHN A. WILKINSON and I. A. GIBSON. *J. Am. Chem. Soc.* 43, 695-700(1921).—Soln. of HCHO (800 or 600 cc.) varying in concn. from 0.29% to 38.4% were distd. from 1-l. round bottom flasks with a Hempel distg. head and Liebig condenser. The original soln., the different portions of the distillate and the residue were analyzed for HCHO and the results are shown by means of curves in which the percentages of HCHO present in the distillates are plotted as ordinates and the cc. of distillates as abscissas. The concn. of HCHO in the distillates obtained from the soln. of higher concn. was always less than in the residue while the solns. of medium concn. (about 8%) distd. over almost as const. boiling mixts. The difference between the concn. of the original and the first distillate decreased up to 8% HCHO , where there was very little difference, but beyond this point the distillate was always weaker than the residue. With solns. of concns. of 0.29 to 10% the residue was weaker than the last distillate, but beyond 10% the residue was stronger. These facts are explained by the fact that HCHO in H_2O forms a polymer

with a lower vapor pressure than the HCHO with which it is in equil. With concns. above 15% HCHO is present chiefly as a polymer.

G. W. STRATTON

An equation of state for saturated and superheated steam between 30° and 180° . H. V. STEINWEHR. *Z. Physik* 3, 466-76(1920).—Levy's (*C. A.* 3, 2892) equation for steam is founded on two assumptions: (1) the applicability of the gas laws for $(\text{H}_2\text{O})_2$ and H_2O , and (2) the independence of the heat of dissociation of the temperature of the reaction. The last assumption is not well grounded and the measurements of the Reichsanstalt show that the heat of dissociation is proportional to the square of the abs. temp. The equation as modified by this assumption fits the data better.

F. C. HOYT

Saturation pressures of some vapors between 10° and -181° . F. HENNING AND A. STROCK. *Z. Physik* 4, 226-44(1921).—Temps. are measured by means of a Pt resistance thermometer that has been directly compared with a H standard, and pressures are measured to 0.01 mm. on a Hg manometer. For the constants b , c , and a , resp., in the Nerst equation $\log p = -b/T + 1.75 \log T - cT + a$ the following values are obtained; CS_2 , 1682.38, 0.0052980, 5.44895; SO_2 , 1561.36, 0.006157, 6.20476; NH_3 , 1393.60, 0.0057034, 5.89654; CO_2 , 1279.11, 0.0020757, 5.85242; HCl , 905.53, 0.0050077, 4.65739; PH_3 , 845.57, 0.0061931, 4.61480; C_2H_6 , 834.13, 0.0083753, 5.32340; CH_4 , 472.47, 0.0096351, 4.60175.

F. C. HOYT

The methods of determining temperature, the means of carrying them out and their importance for obtaining exact results in chemical operations. SCHELL. *Chem. Ztg.* 43, 609(1919).—S. discusses the different thermometric instruments, such as the glass thermometer with a mercury, alcohol or pentane bulb, the Stock steam pressure thermometer, thermo-elements, the resistance thermometer, Seger cones and the optical pyrometer. The thermometer scale of the physical technical imperial institute, which is based on theoretical principles, is represented by a series of fixed points in addition to those of the m. p. of ice and the b. p. of water. These fixed points are the melting or solidification points of Hg (-38.89°), Sn (plus 231.84°), Cd (320.9°), Zn (419.4°), Sb (630°), Ag (960.5°), Au (1063°), Cu (1083°), Pd (1557°) and Pt (1764°); the transition point of Na_2SO_4 (plus 32.38°) and the b. p. of O (normal minus 180°), CO_2 (minus 78.5°), naphthalene (217.96°), benzophenone (305.9°) and S (444.53°). The scale between these fixed points from the m. p. of Hg to the boiling point of S is detd. by interpolation with the aid of a simple formula, by means of platinum resistance thermometer which is tested only at the m. p. of ice and at the b. p. of water and S. Above the boiling point of S, a thermoelement consisting of Pt and 10% Pt-Rh is used. Its thermo range lies between 300 and 1600° and is represented by an equation in the third power, whose consts. are derived from the detd. values at the m. ps. of Cd, Sb, Au, and Pd.

ISMAR GINSBERG

Properties of diagrams. Representative curves of the displacement of equilibrium of chemical systems. JEAN BARBAUDY. *Compt. rend.* 172, 591-3(1921).—Some obvious graphical compn. relations in simple systems are given. E. D. WILLIAMSON

The precipitation of tin by iron. N. BOUMAN. *Univ. Amsterdam. Rec. trav. chim.* 39, 711-14(1920); cf. Kolthoff (*C. A.* 15, 1665).—B. has studied the behavior of Fe and Sn and their salts in aq. soln. from the e. m. f. viewpoint (*C. A.* 15, 351) of Smits, according to which the non-pptn. of Sn by Fe may be attributed to an increased mobility of the Fe surface due to a perturbation of the internal equil. of the surface. This situation can only exist in solns. containing practically only Sn and Fe ions. According to Kolthoff complex formation with HCl diminishes the Sn-ion concn. so much that Fe cannot cause pptn. but B. worked in 2 N HCl soln. and has already shown that his theoretical ideas are confirmed under the conditions used. Some further expts. are described in this paper. To 500 cc. of a SnCl_2 soln. containing 1% Sn 100 cc. 8 N HCl were added. To the clear soln. 100 g. ferrum reductum were added and the whole

boiled. The potentials of the Fe and of the Sn were measured from time to time with a calomel electrode. At the beginning the Fe potential was -505 and that of the Sn -533.5 . After 1 hr. both were -603 and the soln. was nearly free from Sn. The ppt. was washed free from Sn and when dissolved in HCl gave a strong test for Sn. Thiel and Keller (*C. A.* 5, 42) obtained similar results but did not explain them satisfactorily. The statement of Prins in her "Handleiding tot kwalitatief analyse" p. 72, originally questioned by Kolthoff, is correct and the explanation of the phenomenon has been found.

E. J. WITZEMANN

The crystallization of metals by galvanic precipitation and certain related phenomena.

A. H. W. ATEN and MISS LOUISE M. BOERLAGE. Univ. Amsterdam. *Rec. trav. chim.* 39, 720-35(1920).—The galvanic pptn. of metals gives rise to the following forms: (1) well formed isolated crystals; (2) a continuous layer distinctly cryst. in structure; (3) a smooth continuous layer without visible cryst. structure; (4) elongated crystals with needle or aigrette form; (5) black deposit. All these forms may pass from one to the other by imperceptible transitions. Of these forms (1) has been least studied. For the study of the course of crystn. in galvanic pptn. it deserves further attention. The process of deposition is commonly pictured thus: the electrons that the current brings to the cathode pass into the liquid and combine with the metallic ions present to form metallic atoms. The formation of the metal thus takes place in the liquid and it is the supersatd. soln. of this that crystallizes. This crystn. is comparable to all other crystns. and its velocity will be detd. by the rate at which the crystal germs are formed and by the velocity of growth of the crystals, with an additional factor, namely, the change of the polarization tension with the current density, $(\Delta e / \Delta d)$. When the no. of germs is large but the rate of growth small a great no. of small crystals will be formed, and in the contrary case a small no. of large crystals. On electrolyzing a soln. containing no crystals of Ag the soln. when supersatd. seps. cryst. germs on the cathode. New germs are formed and additions to those already deposited occur as electrolysis proceeds. The velocity of this growth will be proportional to the surface of crystal and degree of satn., i. e., $v = kS(C - c)$, where C is the concn. of Ag in the soln., c the concn. of the satd. soln., S the cryst. surface in sq. cm. of the cathode, v the velocity of growth (i. e., the amt. of Ag deposited per sec. on the crystals already present). If d is the current density, d/F gram equivalents of Ag will sep. per sec. per sq. cm. of cathode. The equation $d/F \geq kS(C - c)$ expresses the relations when all Ag sepd. is used in nourishing the crystals already formed, and a large variety of results may be accounted for on the basis of variations in the factors in this equation. Only a small part of the surface of the cathode is utilized and new crystals are not formed. Either the current passes only by means of the crystals present, which accounts for growth only on the crystal faces, or the current utilizes all the cathode surface but the supersatd. soln. no longer gives rise to crystal germs. The 2nd suggestion is considered improbable and the 1st is discussed. The metals that sep. in a microcryst. layer also cover objects in a uniform manner.

E. J. WITZEMANN

Contribution to the knowledge of passivity. Researches on iron and nickel. C. A. LOBBY DE BRUVN. Univ. Amsterdam. *Rev. trav. chim.* 40, 30-64(1921).—This paper is to be followed by another in which the results for Ni and the discussion will be given. d. B. lists 7 types of theories for the state of passivity of which the 1st 6 have been frequently reviewed (cf. Byers, *C. A.* 3, 274; Beunett and Burnham, *Z. Elektrochem.* 22, 377(1916); *C. A.* 10, 1963). The 7th is a theory which rests on the principles of the theory of allotropy of Smits and attributes the phenomena of polarization and of passivity to a delay in the internal equil. between the atoms, ions and electrons in the metallic phase. This theory is deduced from electrochem. considerations of Smits (*C. A.* 8, 1038; 12, 445) and Smits and Aten (*C. A.* 8, 3140; 9, 1140, 3023; 10, 846, 2924, 2829; 11, 904) based on Smits' theory of allotropy (*Z. physik. Chem.* 76, 421(1911)); cf.

C. A. 5, 1861). According to this theory a substance considered simple is really complex and each phase is in a state of equil. between diverse kinds of mols. and their ions. The electrochem. theory is based on the hypothesis that the neutral particles, ions and electrons are present in the liquid phase as well as in the metal or metalloid phase. In each of these phases the constituents are bound by a homogeneous equil., while the corresponding particles are in heterogeneous equil. in the various phases. The heterogeneous equil. between the ions and the electrons both det. the difference of potential and both may be used to deduce an equation relating to the fall of potential. It was shown by Smits and Bijvoet (*Verslag Akad. Wetensch. Amsterdam* 27, 311(1918); cf. C. A. 13, 2477) that the position of a metal in the soln. tension series is detd. by its soly. product, which is equal to the product of the concns. of ions and electrons in the soln. formed when one immerses the metal in a liquid. Thus electrochem. processes may be considered analogous to the reactions between salts with ions of the same name, where the electron takes the place of the ion. In order to explain polarization and passivity of metals it is necessary to admit that the establishment of equil. in the metal can be delayed. Consequently if one carries away some ions and electrons, as occurs in anodic polarization, the surface of the metal may become deficient in these constituents. In this case a change in the soly. product of the metal arises by which it is superficially in a more noble state with a more positive potential. Similarly cathodic polarization leads to a potential displacement of the opposite kind and an increase of the concn. of charged particles in the superficial layer of the metal. If a polarizable metal contains various kinds of ions like Fe this change due to polarization is accompanied by a change in the relative concns. of Fe^{++} and Fe^{+++} ions in the metal. When ennobling occurs the concn. of Fe^{+++} is relatively higher. Experience has shown that H_2 and O_2 ought to be considered as negative catalysts in the establishment of equil. in the metal (Forster, Z. Elektrochem. 22, 95(1916); cf. C. A. 10, 2192). Similarly halogens, especially Cl, have an accelerating action. The influence of Fe ions on the potential of Fe and the influence of H_2 on the potential of a metal susceptible of polarization as detd. by previous work (Smits and de B., *Verslag Akad. Wetenschappen Amsterdam* 25, 901(1916); 24, (?) 270(1917); cf. C. A. 11, 2993) is reviewed. The experimental part of the paper is divided into 2 parts entitled (A) "Equil. potential of Fe. Chemical polarization" and (B) "Fe as anode and cathode." Each part has a no. of subheadings. E. J. WITZEMANN.

Construction and use of alignment charts. A. J. V. UMANSKI. *Chem. Trade J.* 68, 233-6, 269-71(1921).—Chemists have somewhat neglected the use of nomograms as a mechanical help in calcs. A nomogram takes the place of a special slide-rule graduated for a particular purpose. The methods of constructing the scales are given for a number of particular cases, including the adiabatic compression of a gas, evapn. of a solvent to get a given strength of soln., reduction of gaseous vols. to normal temps. and pressure and rate of evapn. for a soln. of known vapor tension in an atm. of different vapor tension. E. D. WILLIAMSON

An inductance coil for use in preparing colloidal suspensions of metals (Cadenhand)
 1. Contributions from laboratory practice (concentration of solutions) (Rudolph) 1.
 Inversion of cane sugar by silicic acid (Mary) 28.

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3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

GERALD L. WENDT

Augusto Righi. *Giorn. chim. ind. ed applicata* 2, 346(1920).—Obituary.

ROBERT S. POSMONTIER

Centers of absorption of colored solutions. EMILIO ADINOLFI. *Atti accad. Lincei* 29, II, 38-41(1920).—In view of the disadvantages attending the usual methods employed to ascertain the centers of the bands of absorption spectra, A. suggests the following procedure, which gives continuous absorption curves. The light, before impinging on the slit of the spectograph, is passed through concns. of the substance, diminishing regularly by diffusion. By means of a capillary funnel the soln. is passed to the bottom of a test-tube contg. the solvent, the tube serving as a lens. The surface of sepn. between solvent and soln. being arranged normally to the slit, the field of the spectograph shows,

prior to diffusion, a sharp demarcation between the part of the absorption spectra and that corresponding with solvent alone. As diffusion begins, the centers of absorption become successively blunted in dependence on their intensity; and become distributed on a curve which is a function of the wave length the intensity of absorption, and the gradient of the concn. Photographic investigation shows that the absorption spectra of K and Ca permanganates exhibit 8 centers of absorption, the third, with the wave length 5255 Ångström units, having the greatest intensity. The 2 absorption spectra are identical within the limits of accuracy of the measurements made. The frequencies of the 8 centers of the visible spectrum are connected by means of the relation $\nu = \nu_m \pm 753n$, where ν_m represents the frequency of the max. of the centers and n the whole numbers from -2 to $+5$. J. C. S.

SODDY, FREDERICK: The Interpretation of Radium and the Structure of the Atom. 4th Ed. revized and enlarged. New York: G. P. Putnam's Sons. 260 pp. \$3.75. For review see *Proc. Am. Soc. Civil Eng.* 47, 563(1921).

4—ELECTROCHEMISTRY

COLIN G. FINK

A survey of American electrochemical development. WALTER S. LANDIS. *Chem. Age* (N. Y.) 29, 163-5(1921). E. H.

New types of electric furnaces. ANON. *Metal Ind.* 19, 149-50(1921).—Two elec. furnaces of the muffled arc type are described, one a 1500-lb. (3300-kg.) unit and the other a 50-lb. (110-kg.) unit. The larger unit forms a balanced steady load on a poly-phase circuit and has electrode regulators automatically maintaining the desired power input. It consists of a steel-plate shell lined with standard fire-clay shapes and a bulged boiler-head bottom. The roof frame is hinged to a bar in the shell at the front of the furnace, the same bar carrying a three-legged cast-Fe spider mounting the electrode-supporting mechanism. The space in the muffle between the electrode and wearing block and the side of the muffle is filled with crushed graphite. The triangular space where the cross electrodes meet in the middle is filled with graphite and tar to insure a good contact. The small unit operates on a single-phase, has a flat hearth and a different electrode and block arrangement. A horizontal C block, imbedded in crushed graphite extends across the bottom carrying a wearing block on each end. Between the wearing blocks are four carborundum bricks which form the hearth. Two graphite electrodes extend through the roof to the wearing blocks. Metal (Zn, etc.), losses are 0.75-1.5%, compared to 3.5-8% for fuel-fired furnaces, while thermal efficiencies are 30-80% compared to 1.5-16%. W. H. BOYNTON

The frequency problem in the steel industry. B. G. LAMME. *J. Am. Inst. Elec. Eng.* 40, 294-300(1921); *Assoc. Iron & Steel Elec. Eng.* 3, 69-84. S. S. WALES. *Ibid* 155-8. BAKTON R. SHOVER. *Ibid* 158-9.—The pros and cons of 25-cycle and 60-cycle. C. G. F.

Cell depolarized by air. C. FÉRY. *Compt. rend.* 172, 317-20(1921).—A modified Le Clanche cell is described. The Zn electrode is placed at the bottom of the jar; it consists of a flat plate. The C electrode is cylindrical in shape and extends the full length of the liquid column, the bottom being placed within a few mm. of the Zn plate. When the circuit is closed the bottom of the C electrode becomes polarized by the deposition of H gas as usual. When the circuit is open this H is caused to go into soln. again at the bottom of the electrode and appears at the top of the rod, where it is removed by the dissolved O₂ of the air. This type of cell is more economical because of the elimination of MnO₂, and has three times the life of the older type. C. R. PARK

Battery charging. H. H. BLISS. *J. Elec. Western Ind.* 46, 520(1921).—A detailed

review. The subject is treated under the following headings: Control of current; using the tungar or the Hg-arc rectifier; hydrometers; battery losses. C. G. F.

The lead storage battery in industrial plants. BECKMANN. *Chem. Ztg.* 45, 171-2 (1921).—Criticizes Langer (cf. C. A. 15, 1657) on the basis that his observations were on a single battery. It is claimed overcharging does not seriously harm the negative plate. Normal charge should be followed by an overcharge at low current to avoid sulfatization and as an aid in bringing all cells up to the same degree of charge. 2.6 volts at $1/2$ normal c.d. should be taken as indicating full charge. B. maintains ordinary hydrometers in use are sufficiently sensitive. M. KNOBEL

Industrial electrical heating. A. E. HOLLOWAY AND H. L. GARBUTT. *J. Elec. Western Ind.* 46, 486-519 (1921).—A detailed review. Elec. ovens for enameling, paint drying, regulating humidity, etc., are described; likewise elec. furnaces for steel and brass making. C. G. F.

An inductance coil for use in preparing colloidal suspension of metals. (CADENHEAD) 1. The crystallization of metals by galvanic precipitation (ATEN, BOERLAGE) 2. Quartz glass and the mercury arc lamp (BERLEMONT) 19.

ROSMAN, L. A. S.: *Nederlandsche electrotechnische kalender voor het jaar 1921*. Amsterdam: Moutgcm & de Does. 568 pp. f3.60. For review see *Chem. Weekblad* 18, 208 (1921).

Storage batteries. GRANT WHEAT. Can. 210,743, Apr. 19, 1921. The battery comprises a casing, battery elements in the casing, conductors in the casing to receive current from the battery elements and charging terminals for the battery on the casing.

Dry cells. ARLE W. SCHORGER. Can. 212,416, June 7, 1921.

Electric batteries. ARTHUR V. WILKER. Can. 210,019, April 26, 1921. In prep. a mix for dry cells adapted to become active on the addition of water a hot soln. of $ZnCl_2$ and NH_4Cl is mixed with pulverulent C and MnO_2 to secure a moist mass. This is dried, leaving the depolarizer impregnated with the electrolyte salt.

Dry batteries. WALTER B. SCHULTE. Can. 211,225, May 3, 1921. A dry battery consists of a set of sep. open-ended vertical compartments in a case, a cell anchored at its upper end in each compartment and spaced from the bottom of the compartment, each cell having a water-repellent wrapper completely encasing it. Cf. C. A. 15, 1464.

Electrolytic cells. JNO. HARRIS. Can. 211,543, May 17, 1921. A cell specifically adapted for producing hydrogen and oxygen has a plurality of cell units each comprising a casing with openings in the opposed side walls thereof, an electrode within each opening and spaced from the wall corresponding thereto with a diaphragm of permeable material covering each electrode.

Apparatus for the electrolysis of solutions. JOHANN K. LANGHARD. Can. 212,191, May 31, 1921.

Electroplating carbon articles. VICTOR C. HAMISTER. Can. 211,899, May 24, 1921. The walls of a hole in a C article are electroplated by continuously flowing a metal-bearing soln. along the walls and passing an elec. current to the walls as cathode from an anode having an active surface located within the hole.

Dry storage batteries. WM. GARDINER. Can. 211,293, May 3, 1921. The electrolyte of a storage battery consists of powdered glass, an acid soln. and a silicate.

Terminal connections for dry cells. HOMER W. JONES. Can. 211,227, May 3, 1921. Structural features are specified for a terminal for the electrode of a dry cell, the upper end of the electrode being below the seal of the container.

Carbon electrodes. P. LEONE TAGLIAFERRI. Can. 211,671, May 17, 1921. An app. for baking C electrodes consists of one or more conducting bars connected to a source of current and extending through the electrode enclosed in an insulating casing.

Electric cells for miner's lamp. THEODORE STRETTON. Can. 210,973, Apr. 26, 1921. The battery comprises a casing, electrodes mounted therein, one disposed within the other, on supports at the bottom of the casing, a central post also integral with the casing, a lid for the casing and means for securing the lid upon the upper end of the central part.

Electrode holders. CARL W. SODERBERG. Can. 212,181, May 31, 1921. An electrode holder has means for permitting a limited independent outward movement of the conductor elements, thus providing for a controlled slippage of the electrode.

Furnace electrodes. CARL W. SODERBERG. Can. 212,421, June 7, 1921. A furnace electrode consists of a perforated mantle having an electrode mass tamped into it and having ribs penetrating into the mass. The electrode may be made in sections in the furnace by tamping the raw mass into the mantle and baking the mass by passing a current through the mantle.

Furnace electrodes. JESSE C. KING. Can. 212,184-90, May 31, 1920. According to these patents furnace electrodes are coated, resp., with a mixt. of finely ground non-oxidizing material and metal particles, with finely ground zirconia with a carbonaceous binder, with finely ground chromite, with finely ground dolomite, with bauxite, and with magnesite mixed with a carbonaceous binder, and with a non-oxidizing material applied to the core and a second coating of cementitious material.

Negative plates for electric accumulators. ADOLFO POUCHAIN. Can. 211,181, May 3, 1921. A layer of celluloid in plastic condition is pressed between two perforated zinc plates to form a negative plate for electric accumulators.

Negative plates for electric accumulators. ADOLFO POUCHAIN. Can. 211,180, May 3, 1921. A zinc plate is reinforced by a metal of high elec. cond. and having a surface which is not attacked by the electrolyte when the circuit is closed.

Protective coating for the active mass of electric accumulator plates. METALLATON, G.M.B.H. Ger. 322,250, Jan. 31, 1918. The surfaces of the plates are provided with a porous metal coating applied by the spraying process. To produce the porous coating there are sprayed on to the plates, with the basic material, which depends on the metal structure of the plate skeleton, other substances of an indifferent nature (e. g., kaolin, clay, infusorial earth, ashes, etc.) or metals or metal alloys, the foreign substances being subsequently rendered inactive or removed.

Galvanic batteries. R. SCHUSTER. Can. 211,196, May 3, 1921. In a galvanic battery having anolyte and catholyte, means for eliminating the products of decomposition of the catholyte comprises an impervious container having a foot plate with a recess and passages laterally extending from the recess and an ascending pipe in communication with the recess in the foot plate.

Diaphragms for galvanic cells and the like. R. SCHUSTER. Can. 211,197, May 3, 1921. The diaphragm for galvanic batteries is so modified in cross-section in its upper part that the passage of the electrolyte can take place more quickly than in the lower part of the diaphragm. This may be effected by the insertion of a second shorter diaphragm or by employing a top piece of less wall thickness.

Press-form for electrodes of galvanic cells. WILHELM FRIEDRICH. Ger. 324,586, Nov. 6, 1919. The press-form is made of cement, gypsum, white-ware material, porcelain, glass or the like and is provided with a metal reinforcement which absorbs the internal pressure. The press-form consists preferably of an outer metal tube, an inner tube of glass, porcelain, quartz, white-ware material or the like, and a hardened mass filling the space between said two parts.

Electrolytic refining of tin. JAS. P. NORRIS. Can. 211,475, May 10, 1921. An electrolyte for refining Sn consists of H_2SO_4 and about 20% of Na_2SO_4 . A small amt. of cresylic acid may be added to prevent undesirable deposits on the cathode.

Electrolytic refining of tin. JAS. P. NORRIS. Can. 211,476, May 10, 1921. An

electrolyte for the electrodeposition of Sn consists of fluosilicic acid with a small amt. of H_2PO_4 to render the Pb content of the impure Sn insol. Cresylic acid may be added to prevent growth of Sn crystals on the cathode.

Electrodeposition of metals upon iron and alloys of iron. ROBERT J. FLETCHER. Can. 211,912, May 24, 1921. The surface of the Fe or its alloy is subjected to an electrolytic cleansing treatment in a bath contg. H_2SO_4 and $CuSO_4$ and the required metallic coating is deposited on the cleansed surface.

Material for calcium carbide production. S. A. WISDON. Can. 212,241, May 31, 1921. Part of the free water is removed from the residue in the production of C_2H_2 from CaC_2 and partially dried product is mixed with coke which absorbs the remaining water of the sludge. The mixt. is used as aggregate in the production of carbide.

Desulfurizing iron sulfide by heating. LEWIS THOMPSON WRIGHT. Ger. 322,286, Nov. 23, 1912. An elec. current passed through the FeS heats it to such a degree that all of the S is driven out and metallic Fe is produced. Into a tilting elec. furnace, with inclined lining and a charging door in the removable cover, is put a charge of fresh pyrites or of partly desulfurized pyrites. The furnace is equipped with an adjustable electrode, which passes through the cover of the furnace. The current passes through the charge either directly or in the form of an arc, and then through the bottom of the furnace. The cover has an opening connected with the condenser, through which opening the S vapors are discharged and condensed. Samples of the molten mass are drawn from time to time, and thus the progress of the process can be supervised. Finally the slag is removed and the charge of pure Fe poured into a mold, whereupon a fresh charge of pyrites is put into the furnace.

Electrolytic soft iron. MAX SCHLOTTER. Ger. 308,543, Feb. 7, 1918. To reduce the ferric salts formed by electrolysis there is added to the electrolyte hydroxylamine, hydrazine, or compns. thereof, or C-free electrolytic Fe in the form of shavings or the like in the electrolyzing chamber or in a sep. reduction chamber.

Electrolytic treatment of organic substances. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. Ger. 310,023, Oct. 2, 1916. Without employing a diaphragm, electrolyte solns. of different sp. gr., one of which will keep the substance to be treated in soln. are introduced into the process in continuous operation. The passage of the org. substance to the opposite pole is prevented by causing electrolyte soln. to flow from the direction of the opposite pole toward the active pole.

Electrolysis of aqueous solutions with electrodes capable of being attacked, such as iron electrodes. NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB. Ger. 321,771. June 27, 1919. The electrolysis is conducted in presence of a sol. silicon compd., sol. silicates being added to the electrolyte. Such addition is preferably effected in small amts. at relatively long intervals. This process is applicable to the electrolysis of nitrate or nitrite solns. to produce NH_3 and soda lye. While in the electrolysis without waterglass, the tension of the bath is 3 v., the attack of the electrodes is several mm. a year, the loss of N up to 60%, and the current utilization of the bath less than 50%, the electrolysis with 0.5% of silicic acid added to the electrolyte showed a bath tension of 2.2 volts, electrode attack hardly measurable, N loss about 5%, and current utilization of bath about 90%.

Perborates. JOHANN K. LANGHARD. Can. 211,913, May 24, 1921. Perborates are produced by the electrolysis of a borate soln. in the presence of a cyanogen compd.

Aluminium nitride. MARK SHOELD. Can. 210,713, April 19, 1921. A mixt. of briquets contg. finely divided aluminous and carbonaceous material in the proportion of 3 to 1 by wt. and larger substantially infusible resistor-elements which do not enter into the chem. reaction and which are of greater elec. cond. than the briquets and prevent fusion of the briquets, is subjected to a N-contg. gas and to the passage of an elec.

current, which heats the briquets by the heat developed in the resistor-elements and changes the briquets to AlN. cf. C. A. 14, 2532.

Apparatus for the electrolysis of anhydrous magnesium chloride. EDGAR A. ASHCROFT. Can. 212,269, June 7, 1921.

Electrolytic cells. O. H. JEWELL. Can. 211,902, May 24, 1921. Structural features are specified for making NaOH and Cl from brine. Cf. C. A. 15, 211.

Electrolytic cells. WM. M. JEWELL. Can. 211,901, May 24, 1921. The cell specified is for the production of Cl and caustic alkali and H. A porous diaphragm is used. Cf. C. A. 15, 211.

Electrolytic apparatus. GEO. O. SEWARD. Can. 212,126, May 31, 1921. The app. specified is for the production of a metal lighter than the electrolyte.

Electrolytic apparatus. ISAAC H. LEVIN. Can. 210,570, May 17, 1921. Structural features of app. of the filter-press type are specified.

Plating apparatus. GERHARD T. POTTHOFF. Can. 211,589, May 17, 1921. The app. comprises a container and means for rotating the container a number of revolutions in one direction and then rotating it a number of revolutions in the opposite direction. One of the electrodes is disconnected from the source of current when the container is not in motion.

Separators for electrolytic cells. ISAAC H. LEVIN. Can. 211,569, May 17, 1921. The separators specified provide for the free circulation of the electrolyte to and from the compartments on opposite sides of the diaphragm and to and from the filling devices in cells used for the production of O and H.

Indicators for electrolytic cells. ISAAC H. LEVIN. Can. 211,568, May 17, 1921.

Electrodes. ISAAC H. LEVIN. Can. 211,567, May 17, 1921. An electrode consists of two sheet-metal plates having oppositely disposed depressions adjacent to the top and a flat metal terminal bar of substantially the same width as the depressions placed between them and welded to them.

Water-feed attachment for electrolytic cells. ISAAC H. LEVIN. Can. 211,566, May 17, 1921. The fill cup shown may be utilized to prevent a blow out in the event of the development of excessive pressures in the cells.

Electrolytic cells. ISAAC H. LEVIN. Can. 211,565, May 17, 1921. Structural details are specified whereby a gas-tight insulating support is afforded the terminals of the electrodes.

Electrolytic apparatus. ISAAC H. LEVIN. Can. 211,564, May 17, 1921. The app. comprises cells of the unipolar type with electrodes of large surface area and off-take system common to all the cells.

Apparatus for subjecting substances to the electrostatic stress of a high tension electric discharge. F. G. NICH and R. M. LAGGITT. Can. 211,484, May 17, 1921. Structural features.

Apparatus for electroplating. GERHARD T. POTTHOFF and KURT T. POTTHOFF. Can. 211,489, May 17, 1921. The app. has in combination with the tank, the soln., an elec. current and a conveyor, a series of tapering spindles carried by the conveyor, each spindle being adapted to enter the end of a tube to be plated and support it in a horizontal plane and a device to engage and detach the tubes in succession.

Electrolytic apparatus. CECIL M. WALTER. Can. 211,877, May 24, 1921. Electrolytic app. for detinning processes comprises a fixed vat for electrolyte and a rotatable perforated drum for the material under treatment having cathode plates radially and longitudinally disposed therein with permeable means for preventing the material from contacting with the plates. The plates and permeable means divide the drum into compartments.

Electric furnaces. JAS. H. GRAY. Can. 212,250, May 31, 1921. The openings in

the furnace roof through which the electrodes pass are shaped to permit the tilting of the furnace without movement of the electrodes.

Heating by electricity. E. H. JONES and ALLOY WELDING PROCESS LTD. Brit. 157,534, Oct. 14, 1919. Mineral or manufd. graphite, or gas or retort C is incorporated in an electrode, welding rod, or soldering stick to provide the required proportion of C in the metal when fused. A mixt. of two or more of the above mentioned materials may be used, and they may be applied in powder or paste form. The material may form an ingredient of a slag-forming electrode covering and may be incorporated with other alloying materials. The proportion of graphite or the like in the electrode may be 0.3% when Ni steel is to be deposited, and 4% in the case of cast iron.

Calcium carbide. F. M. BECKER. Can. 212,217, May 31, 1921. A mixt. of hydrated lime and non-coking carbon in proper proportions is compressed, heated under non-oxidizing condition to drive off the combined water and harden the mixt. which is then suitable for making carbide. Cf. C. A. 14, 698.

Filaments from tungsten or the like. The WESTINGHOUSE METAL FILAMENT LAMP CO., LTD. Ger. 322,303, Apr. 26, 1914. Prior to the mechanical treatment, or between the different phases of the same, the blanks are subjected to the action of vapors or gases of S, Se, Te, or compds. thereof, such as H_2S , H_2Se or H_2Te , which produce on the blanks deposits capable of being afterwards completely removed, or they are surrounded with a fused mass of S, Se, or Te which can subsequently be readily removed. This prevents oxidation of the blanks and facilitates the rolling and drawing processes without harmfully affecting the material of the blanks. The gases or vapors may be used in heated state, so as to cause at the same time the heating of the blanks. Before the treatment, or in the course of the same, the blanks may be exposed to the flame of combustible compds. of S, Se, or Te.

Filaments of graphite-like carbon. SIEMENS & HALSKE AKT.-GES. Ger. 322,302, Mar. 29, 1914. C is mixed with a metal or metal compd. and then heated for such a period and to such temps. that a mass will form which is in a certain degree ductile and pliant. C is preferably applied to a filament or rod of W, whereupon the body is heated in an elec. furnace to a temp. in the vicinity of the m. p. of W or above until a homogeneous mass has formed which is to a certain degree ductile and deformable. The body obtained by heating is further condensed and homogenized by high pressure or mechanical treatment. The heating is conducted in absence of air, preferably in a C tube surrounded by C.

5—PHOTOGRAPHY

LOUIS DERR

Use of oxidizers, especially quinones, in chlorinating and brominating silver in silver images. A. AND L. LUMIERE AND A. SEYEWETZ. *Bull. soc. franc. phot.* [3] 7, 267-70(1920).—Benzoquinone or its sulfonic derivative, in presence of HCl or HBr, supplies a new method of chlorinating or brominating the Ag without adding anything to the image except Cl or Br. Baths are: water 1000 cc., quinone 5 g., NaCl 6 g. or KBr 11 g., H_2SO_4 3 cc.

L. DERR

Eder-Hecht neutral-wedge photometer for sensitometry and light-measurement. J. M. EDER. *Phot. Korr.* 56, 244-71(1920); 57, 1-9, 41-4, 83-6(1921).—The essential part of the instrument is a Goldberg neutral wedge, 8×12 cm., covered with a transparent scale. The source of light is a standard Hefner lamp, exposure-time 1 min. at 1 m. distance; or Mg ribbon may be burned in an alc. lamp at 3 m. Many tables of calibrations and tests are given.

L. DERR

Inverse images, colored by imbibition. J. I. CRABTREE. *Bull. soc. franc. phot.* [3] 8, 27-30(1921).—The gelatin vehicle of the Ag image is dyed with methylene blue

methylen green, or any other dye easily reduced to its leuco-base, and the plate is then immersed in an acid fixing bath, which destroys the color on the Ag in proportion to the image-density at the particular point. The plate is then washed and the Ag dissolved out by any standard reducer; there remains an image in color in the gelatin, positive if the Ag image was a negative and conversely. The theory is as yet obscure.

L. DERR

Extinction by a blackened photographic plate as function of wave length, quantity of silver, and size of grains. A. DRUMMENS. *Proc. Acad. Sci. Amsterdam* 23, 848-66 (1921).—The blackening was measured by a Moll compensating thermoelectric extinction meter, and the blackening ratios were found to be a function of the wave length of the incident light, in agreement with previous work. Plates developed with glycine are satisfactory as light absorbers for the yellow-green region, but all plates should be tested for absorption in other spectrum regions. The quantity of Ag per unit of plate area was found not proportional to the blackening, the difference from accepted results being attributable to unequal size of grain in the different deposits. With attenuated deposits on plates developed with hydroquinone the diameter of individual grains was not measurable; with denser deposits it was 0.49μ . On plates developed with glycine the grain size was fairly constant, averaging 0.96μ .

L. DERR

Color-sensitizing properties of a new series of cyanine coloring materials. A. AND L. LUMIÈRE AND H. BARNIER. *Bull. soc. franç. phot.* [3] 7, 182-4 (1920).—Cyanine A is obtained by starting with a mixt. of dimethylaminoquinoline ethiodide and toluquinaldine ethiodide. It contains 1 dimethylamino auxochrome group. Its plate-sensitiveness curve contains maxima in the blue, yellow, and orange. Cyanine B starts from a mixt. of dimethylaminoquinoline ethiodide and dimethylaminoquinaldine ethiodide. It contains 2 dimethylamino auxochrome groups, and its sensitiveness curve has a deep minimum in the green, with maxima in the blue, yellow, and orange-red, as far as the end of the visible red. Pantochrome is obtained by the condensation of dimethylaminoquinaldine ethiodide with dimethylaminobenzaldehyde. This is a remarkable sensitizer for the whole spectrum, having only a slight fall in the green.

L. DERR

Analysis of photographic emulsions. A. B. HITCHINS. *Bull. soc. franç. phot.* [3] 7, 228-34 (1920).—Detailed directions, for which the original paper should be consulted, are given for detg. the quantity of gelatin and Ag halides in photographic films and obtaining the proportion between the AgBr and AgI.

L. DERR

Improvements in methods for simultaneous development and fixing of photographic images. A. AND L. LUMIÈRE AND A. SEYEWETZ. *Bull. soc. franç. phot.* [3] 7, 234-8 (1920).—The following bath is suggested: water 1000 cc., anhydrous Na_2SO_4 32 g., chloranol 6 g., NaOH 5 g., hypo 60 g. The NaOH may be replaced by 140 g. Na_2PO_4 if 48 g. hypo are used instead of 60 g. Another bath is: water 1000 cc., anhydrous Na_2SO_4 32 g., metoquinone 6 g., Na_2PO_4 100 g., hypo 40 g. These baths give images entirely comparable to those obtained by the usual sep. development and fixing, but require double the normal exposure and 20-25 min. immersion. The first mentioned bath is available for chlorobromide papers, in 2-min. development and double exposure. Inspection is not necessary during the development of plates and this feature may be sometimes useful, as over-development is not greatly to be feared.

L. DERR

Photographischer Notizkalender 1921. M 8.20. Das Photographieren mit Blitzlicht by SCHMIDT. 2nd ed. M 8.20, bound M 11.20. Die Selbstbereitung von Bromöl-druckfarben. 2nd Ed. M 2.40. Der Gebrauch der Blende in der Photographie by H. v. CLES. M 4. Die Spiegelreflexkamera by MAYER AND HANNEKE. M 4.20, bound M 6.70. Halle a. Saale: Wilhelm Knapp. For review see *Papier-Zig.* 46, 1655 (1921).

6—INORGANIC CHEMISTRY

H. I. SCHLESINGER

The binary system: $\text{Ag}_3\text{S}-\text{Ti}_2\text{S}$. HANS HUBER. Univ. Göttingen. *Z. anorg. allgem. Chem.* 116, 139-40(1921).—Thermal analysis of the system $\text{Ag}_3\text{S}-\text{Ti}_2\text{S}$ showed a eutectic at 306° and 55% Ti_2S , while at about 358° the sepd. Ag_3S reacted with the melt to form crystals of $\text{Ti}_2\text{S}_4\text{Ag}_3\text{S}$. The transformation of Ag_3S at 175° could not be detected in mixes with more than 31% Ti_2S .

M. R. SCHMIDT

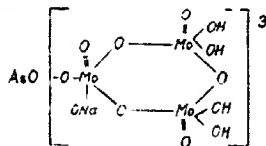
A hitherto unknown copper aluminate of the spinel type. J. A. HEDVALL AND JOSEF HEUBERGER. Örebro Tech. School. *Z. anorg. allgem. Chem.* 116, 137-8(1921).—Mixts. of CuO and Al_2O_3 in the mol. proportions of 1:1, 1:2, and 2:1 heated with 6-10 times their wt. of KCl at about 850° for 1 hr. react easily to form copper aluminate, but on account of a secondary reaction heating without flux to 900° proved more satisfactory, giving a chocolate-brown powder, which appeared under the microscope to be approx. homogeneous in the 1:1 mixt., but showed excess of one or the other reagent in the others. The prepn. was purified by digesting with hot dil. HNO_3 , and was analyzed by splitting with KHSO_4 , showing the compn. to be copper aluminate, $\text{CuO} \cdot \text{Al}_2\text{O}_3$. Larger crystals may be obtained by heating the purified compd. for 0.5 hr. in fused KCl .

M. R. SCHMIDT

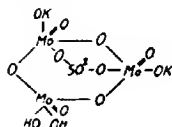
A method for producing dry ammonia. H. W. FOOTE AND S. R. BRINKLEY. *J. Am. Chem. Soc.* 43, 1179(1921).—By passing dry NH_3 into a bottle containing NH_4CNS , the satd. solu. obtained at 0° and atm. pressure contains about 45% NH_3 . By placing the bottle in a water bath, and keeping at room temp. or slightly above, the NH_3 can be drawn off as desired.

G. W. STRATTON

Classification and constitution of the complex derivatives of molybdic acid. L. FORSEN. *Compt. rend.* 172, 631-4(1921); cf. preceding abstract.—The complex acids can be grouped in 3 general series whose formation is indicated by the following examples: (I) Complex orthomolybdic acids $\text{H}_4(\text{Mo}_3\text{O}_{12}) + \text{H}_2\text{AsO}_4 = \text{H}_3(\text{AsOMo}_3\text{O}_{11}) + 3 \text{H}_2\text{O}$; (II) complex metamolybdic acids $\text{H}_4(\text{H}_4\text{Mo}_3\text{O}_{12}) + \text{H}_2\text{AsO}_4 = \text{H}_3(\text{H}_4\text{AsOMo}_3\text{O}_{11}) + 3 \text{H}_2\text{O}$; (III) complex lutcomolybdic acids $3\text{H}_4(\text{Mo}_3\text{O}_{12}) + \text{H}_2\text{AsO}_4 = \text{AsO}[(\text{H}_4\text{Mo}_3\text{O}_{11})_3\text{H}]_3 + 3 \text{H}_2\text{O}$. The acid formed in III is tribasic in its ordinary salts. Its sodium salt has the following structural formula:



"All the known facts relative to the quantity of water of constitution of the complex compds. of the molybdic acids are in accord with the proposed formulas." F. has prepd. a new salt of sulfato-ortho-molybdic acid. Its most probable constitution is:



D. MACRAE

Classification and constitution of the derivatives of molybdic acid. L. FORSEN. *Compt. rend.* 172, 215-7, 327-30(1921).—All known molybdates can be derived from the

two acids: molybdic acid H_4MoO_{12} and metarmolybdic acid $H_4Mo_{12}O_{36}$. In order to explain the formation of anhydrides F. assumes 4 different spacial representations of this acid, depending on two possible arrangements of the -OH groups attached to each Mo atom. They may have the *trans* arrangement on all 3 atoms, the *trans* on two and *cis* on one, the *trans* on one and the *cis* on two, or *cis* on all 3 atoms. In general the proposed formula permits the prediction of stereoisomers, *cis* and *trans*, and optical isomers. It is probable that the enormous increase in the rotatory power of certain org. acids when they combine with compds. of molybdic acid is due to the existence of optical isomers which the preceding theory supposes. II. The pure anhydrous acid $H_4Mo_6O_{18}$ can be prepared. Its anhydrous salts absorb water in soln. and possess all the properties of derivs. of molybdic acid $H_4Mo_6O_{18}$. The tetramolybdates $M_2O_4MoO_4$ aq. and the octamolybdates $M_2O_8Mo_2O_8$ aq. are derived from another anion. The difference in the anions of tri- and tetramolybdates is apparent when they are treated in soln. with HCl. In the resulting acids the ratio of Mo to Na is the same but they are acid or alkaline to helianthine according to whether they are derived from tri- or tetramolybdate. The tetra- and octamolybdates are salts of metarmolybdic acid. The anhydrous tetramolybdates, e. g., $(NH_4)_4Mo_{12}O_{36}$ and $Rb_4Mo_{12}O_{36}$, correspond to the complete anhydride of the above acid. Structural formulas for the molybdic and metarmolybdic acid are suggested.

D. MACRAE

The oxidation of arsenious acid by nitric acid in the presence of mercuric ions (KLEMEENC, POLLAK) 2. A contribution to the solubility problem [ammines] (EPHRAIM) 2.

7—ANALYTICAL CHEMISTRY

WILLIAM T. HALL

The significance of the "specific refraction" for analytical chemistry. N. SCHOORL. Univ. Utrecht. *Rec. trav. chim.* 39, 594-9 (1920); cf. C. A. 14, 3380.—The use of the b. p., the d. and the index of refraction as sufficient data for the identification of a compd. is of limited value for 2 reasons: (1) The purity of the compd. is never perfect and (2) the accuracy of the measurements is limited. The relation between the chem. constitution of compds. and the numerical value of sp. characteristic magnitudes is at present incompletely known, so that it is not possible to deduce the chem. constitution from such numerical values. There is one magnitude, ordinarily called sp. refraction, which is const. for varying densities and is, therefore, independent of temp. and pressure. The simplest form of this is $(n-1)/d$ (Gladstone and Dale, 1854). This property permits a probable identification of a substance (at least of an org. compd.) without requiring any data except the index of refraction and the d. The fact that the sp. refraction for a given compd. can be calcd. is an advantage, but the values lie roughly between 0.300 and 0.600 and this makes its distinctive value small unless the no. of possibilities is small. If the choice becomes greater the method no longer gives a probable identification. This latter statement is discussed and illustrated in 4 paragraphs.

E. J. WITZEMANN

Titration with the hydrogen electrode. W. D. TREADWELL AND L. WEISS. *Helvetica Chim. Acta* 3, 433-46 (1920); cf. C. A. 15, 1262, 1263.—The authors describe a H electrode the change in potential of which can be measured accurately in acidimetric titrations by direct connection to a millivoltmeter. Examples are given showing the titration of strong and weak acids and the detn. of strong acids in the presence of weak acids. The equil. const. of weak acids is calcd. from the drop in potential during the titration.

F. W. SMITHER

Contamination of much used reagents. J. WOLFF. *Z. deut. Oel-Fett. Ind.* 41, 195-6

(1921).—Analysis of a 37.8% NaOH soln. revealed the presence of 1.1% NaClO_3 , which made it unfit for N detn. by the Kjeldahl method. Anhydrous Na_2SO_4 used for drying fatty acids in either soln., contained up to 7% H_2O and one sample contained 1.2% Na_2CO_3 ; it should be dehydrated before use. CaCl_2 (for drying purposes) has frequently an alk. reaction and must first be neutralized by passing CO_2 through the CaCl_2 tubes when used in the absorption train for CO_2 detns.

P. ESCHER

The commercial analysis of salitre. Procedure employed by the official assayers. ANON. *Caliche* 1, 201(1919).—The com. analysis includes the following, H_2O -insol., Cl , SO_4 and ClO_4 . The nitrate is obtained by difference.

C. L. BURDICK

Direct determination of total nitrate nitrogen in nitrates and nitric ethers. I. NORDEN FLYCHT. *Caliche* 1, 210(1919).—Improvements in the method of Devarda and application to the detn. of nitrate nitrogen in powder, nitro explosives, nitrate ores, etc. The materials used have been simplified to standard app. and the procedure has been simplified to give greater accuracy and rapidity.

C. L. BURDICK

The determination of nitrous fumes in air, with special reference to fuse igniters. JAMES MOIR. *J. S. African Assoc. Anal. Chem.* 4, 3-7(1921).—The NO_2 in air can be detd. by reduction to NH_3 in alk. soln., but the results are inaccurate when less than 0.1% of NO_2 is present. Reduction in acid soln. gives low results. For very minute quantities of NO_2 the colorimetric method depending upon the formation of a pink color with α -naphthylamine also fails to give satisfactory results. The most sensitive test appears to be iodo-starch paper. If more than 0.1% of NO_2 is present in the air, the reaction with the test paper is instantaneous. If W represents mg. of NO_2 present in 100 liters of air, and T the time in sec. required to give a brown line with iodo-starch paper, the formula $(W-2)(T+1) = 180$ holds approx. A new test for nitrites or nitrous acid is similar to that of Ilosvay but p -nitroaniline and α -naphthol are used in place of sulfanilic acid and naphthylamine. Prepare soln. A by dissolving 1.5 g. of p -nitroaniline in 40 cc. of concd. HCl and dilg. to 500 cc. Soln. B contains 2 g. α -naphthol, 1.1 g. NaOH and 10 g. AcONa in 500 cc. Mix the liquid contg. a trace of nitrite with from 1 to 10 cc. of soln. A (10 cc. is enough for 0.02 g. NO_2 or 0.015 g. NaNO_2) and heat to 50° until complete diazotization takes place. The product, p -nitrobenzenediazonium chloride, is not decomposed below the h. p. of water. Add the same quantity of soln. B as was used of soln. A. If nitrite was present, an orange-red ppt. of p -nitrobenzeneazo- α -naphthol is formed quantitatively. Filter into a Gooch crucible after 1 hr. 293 pts. of ppt. = 92 pts. of NO_2 or 60 pts. of NaNO_2 . The dyestuff dissolves in NaOH with an intense blue-violet color which may be used for a colorimetric test. Fuse igniter fumes.—The British Govt. requires that the fumes produced shall not give rise to more than 0.5 g. NO_2 from 100 g. of fuse. With a 10-liter bottle, only 1 g. of powder can be taken for the test and the smoke may contain nitrate and carbonate as well as about 1 g. of CO_2 . As soon as water is added to such a mixt., nitrate and nitrite are formed from the NO_2 which was present in the fumes. The distinction between nitrite in smoke and NO_2 can only be made by means of the acidity of the latter. The following 2 methods have been found fairly satisfactory. (1) Keep the gas in contact with neutral H_2O_2 soln. for 24 hrs., then add methyl orange and titrate with 0.1 N NaOH . $2 \text{NO}_2 + \text{H}_2\text{O}_2 = 2 \text{HNO}_3$. Use the empirical factor 1 cc. 0.1 N $\text{NaOH} = 0.0060$ g. NO_2 . (2) Treat the fumes with KI soln. (1 g. in 20 cc.) for at least 6 hrs. with occasional shaking and having the walls of the container well wetted with soln. Titrate the liberated I_2 with 0.1 N thiosulfate; 1 cc. of this = 0.0051 g. NO_2 instead of the theoretical value.

W. T. HALL

Report on special study of the Kjeldahl method. H. W. DAUDT. *J. Assoc. Official Agr. Chemists* 4, 366-73(1921).—In the Kjeldahl method for N the retention of NH_3 by Hg makes necessary the pptn. of the Hg as sulfide. For this purpose $\text{Na}_2\text{S}_2\text{O}_4$ may be

used in place of the more expensive K_2S . $CuSO_4$ is not so effective a catalyzer as Hg but if present in amts. not exceeding 0.5 g. causes no retention of NH_3 . 0.2 g. of Hg or HgO is not sufficient for the hydrolysis of refractory substances and the proportion of acid to alkali sulfate is important. With 10 g. of K_2SO_4 or 8.2 g. of Na_2SO_4 , 35 cc. H_2SO_4 will not give complete decompn. 20 cc. is the most effective vol. of H_2SO_4 , although where considerable carbonaceous matter is present 25 cc. can be used to advantage. With 20 cc. H_2SO_4 , 8.2 g. Na_2SO_4 and 10 g. K_2SO_4 can be substituted for each other. The length of the period of digestion depends on the proportion of reagents and on the nature of the sample. Unless the time is definitely known for the particular substance, a period of 2 hrs. or more should be employed for the digestion, counting only the time of brisk boiling.

S. G. SIMPSON

New method for determination of minute amounts of bromine. ERNST OPPENHEIMER. *Arch. exp. Path. Pharm.* 89, 17-28(1921).—The method advocated is colorimetric. To solns. of known Br content and to the unknown soln. increasing amts. of a standardized Cl_2 soln. (prepd. from $Ca(OCl)_2$) are added in the presence of fuchsin and H_2SO_4 . The color changes, from violet to yellow, are compared and the amt. of Br present in the test soln. is calcd. Cl_2 liberates Br from HBr and bromo-fuchsin is formed.

G. H. S.

Preliminary report on the estimation of hyposulfites and sulfoxylates. EDWARD L. HELWIG. *Am. Dyestuff Reporter* 7, 11, No. 15 12-3(1920).—The method of analysis is based upon the decolorization of an ammoniacal Cu soln., which is reduced to cuprous salt. Thus with hyposulfite. $2 CuSO_4 + 4 NH_3 + Na_2S_2O_4 + 2 H_2O = Cu_2SO_4 + Na_2SO_3 + (NH_4)_2SO_4 + (NH_4)_2SO_4$. The standard copper soln. contains 100 g. $CuSO_4 \cdot 5H_2O$ and 20 cc. concd. NH_4OH in 2 liters. *Analysis of anhydrous $Na_2S_2O_4$ (Lykopon).*—Place 50 cc. of standard Cu soln. in a 150-cc. Erlenmeyer flask and pass 2-3 bubbles per sec. of washed CO_2 into the flask during the titration. Add from a weighed tube, small portions of hyposulfite until the blue color is discharged and det. the wt. required by reweighing the tube. If too much hyposulfite is added, metallic Cu will be pptd. 1 g. Cu = 13.68 g. of $Na_2S_2O_4$. *Analysis of basic Zn sulfoxylate formaldehyde (Formopon extra).*—Dissolve 5 g. of sample in 25 cc. of NH_4OH , dil. to 100 cc., filter, reject the first portion of filtrate and use 10 cc. for the analysis which is carried out precisely as with formopon. *Analysis of Na sulfoxylate formaldehyde (Formopon).*—Dissolve 5 g. in water and dil. to 100 cc.; pipet off 10 cc. into a 150-cc. Erlenmeyer flask and introduce a moderate stream of CO_2 . Add standard Cu soln. from a buret, as long as it is decolorized. Then heat over a free flame and continue adding standard Cu soln. until a faint blue color is not discharged by 10 sec. boiling, 1 g. Cu = 1.2 g. formopon = 1.40 g. formopon extra.

W. T. HALL

Methods for estimating limits of chlorides in chemicals. L. F. KEBLER AND W. H. HEATH. *J. Assoc. Official Agr. Chem.* 4, 360-4(1921).—In expressing limit tests of impurities in chem. reagents such terms as "opalescence" and "turbidity" are vague and are sometimes misleading. In testing for chlorides K. proposes to compare in Nessler tubes with standards prepd. from 0.01 N HCl, and sets arbitrary limits for the various terms used. Methods are given for prepg. samples for testing in the individual cases of about 50 common reagents.

S. G. SIMPSON

The detection of lactic acid. L. HARTWIG AND R. SAAR. *Chem. Ztg.* 45, 322(1921).—A characteristic test for lactic acid has been found to be as follows: To 0.2 cc. of the substance to be tested add 2 cc. of concd. H_2SO_4 . Heat 2 min. on the water bath, cool under the tap, and add 1/2 drop of 5% alc. soln. of guaiacol. The presence of lactic acid is shown by a deep red coloration. This test is sensitive only when no more than 0.2% lactic acid is present. A negative test, therefore, does not necessarily show the absence of the acid unless from previous expt. the percentage is found to be in the vicinity of and somewhat less than 0.2%. To insure this, the substance may be treated with a

solvent for any lactic acid present and the desired approx. concn. prepared from the weighed ext. In *detecting lactic acid in baking powder* the procedure is as follows: In a mortar take up 3 g. of the baking powder in as little H_2O as possible. After the CO_2 has escaped, add methyl orange and make slightly acid with H_3PO_4 . Mix the liquid with sand and plaster of Paris to a pasty consistency and allow to stand several hrs. After the mass has hardened, pulverize it and ext. in a Soxhlet app. with Et_2O . Filter the ext., evap. the Et_2O and dry in the hot-closet. Weigh and by proper diln. prepare a 0.2% soln. Filter through a hardened filter and test as above. The color may be detected in the presence of other org. acids.

S. G. SIMPSON

The determination of chlorine in organic substances; possible losses and their avoidance. A. WERTZEL. *Arb. Reichsgesund.* 52, 635-49 (1920).—If the Cl content of an org. substance is detd. by the analysis of the ash, it is possible that the results will be too low owing to loss of HCl or of alkali chloride during the combustion of the substance. W. in 1917 has shown that it is possible to det. the halogen content of org. substances without burning them. His method consists essentially in extg. the halogen by treatment with 25% HNO_3 or with 10% KOH and detg. the halogen by the Volhard titration method. In this paper the results obtained in about 125 comparative tests are given. The materials analyzed were for the most part org. tissues and the Cl content ranged from about 35% NaCl to 0.35% NaCl. The results show that simple ignition in a Pt or porcelain dish invariably causes loss, sometimes as much as 30% of the total Cl. If, however, the mass in the dish is covered with 1/10 its wt. of CaO and then heated very carefully, as recommended by Nencki and by Schoumow-Simanowski, there is but little if any loss of Cl. It is rather better, however, to rub up the substance and CaO with a little water, to evap. the paste on the water bath and then heat carefully. Heating with a mixt. of Na_2CO_3 and KNO_3 was not so satisfactory.

W. T. HALL

A volumetric method for the determination of the total sulfurous acid in organic substances after the distillation process. VICTOR FROBOSSE. *Arb. Reichsgesund.* 52, 657-69 (1920).—The method is suitable for the analysis of org. materials of various sorts, such as sulfite pulp, bleached gelatin, dried apples or wine. It consists in distg. with 50 cc. of 25% H_3PO_4 , catching the distillate in a measured vol. of standard $NaHCO_3$ soln., oxidizing the sulfite to sulfate by means of 30% H_2O_2 and finally titrating the remaining carbonate with 0.1 N HCl using methyl orange as indicator. After the titration the sulfate may be detd. gravimetrically as a check. Place about 400 cc. of the soln. to be tested, which should preferably contain about 80 mg. of SO_2 , in a 750-cc. flask provided with a two hole rubber stopper. Through one hole introduce CO_2 gas and insert through the other hole a vertical distg. tube 40 cm. long. Then connect with a condenser and lead the distillate into a 300-cc. Erlenmeyer flask contg. about 40 cc. of standard $NaHCO_3$ soln. Boil the contents of the flask 75 min., when about 200 cc. of liquid should have distilled over. To the distillate add a suitable quantity of H_2O_2 and boil about 3 min. until on shaking there is no further evolution of CO_2 or O_2 . Cool, add methyl orange and titrate. In dilg. the original soln. it is necessary to use water that has been boiled with CO_2 passing through it to remove dissolved O_2 . The CO_2 during the distn. is not needed to prevent oxidation but helps to expel the SO_2 , thus shortening the time required. There is likely to be a slight oxidation of SO_2 by dissolved O_2 . Thus in expts. with known quantities of Na_2SO_3 the results were a little low but the oxidation was proved to take place not with SO_2 vapors but only when the SO_2 and O_2 were both in the dissolved condition. The use of a long distilling tube served to prevent appreciable amts. of acids such as acetic from being carried over. The result obtained by this method appears to be somewhat better than those of the iodometric distn. method of Haas, but the method is unreliable for the detn. of traces of SO_2 , as a slight consumption of $NaHCO_3$ may be caused by some other acid.

W. T. HALL

Rapid estimation of aluminium sulfate in alum. P. W. CONWISS. *Paper* 26, 187-9 (1920).—Dissolve 0.6 g. of alum in 25 cc. of H_2O and titrate with 0.1 *N* NaOH in boiling hot soln. until a pink color produced by 2 drops of alkali is not discharged by boiling 1 min. 1 cc. of 0.1 *N* NaOH = 0.00170 g. Al_2O_3 present as alum.

H. H. HARRISON

The determination of hydrogen peroxide by means of stannous chloride. SVEN HÄSSLÉSKOG. *Svensk Farm. Tid.* 25, 149-50 (1921).—The method for the detn. of H_2O_2 proposed by Bertalan (*C. A.* 10, 2565) is not reliable. There is not a single simple reaction involved as claimed. Two other hy-reactions occur, namely: $SnCl_2 + xH_2O_2 \rightleftharpoons SnO_2 \cdot xH_2O + Cl_2 + (x - 2) O$ (see Zsigmondy: *Kolloidchemie*, 2nd Ed.) and $2HCl + H_2O_2 \rightarrow 2H_2O + Cl_2$

A. R. ROSE

The use of sodium persulfate in analysis. L. DÉBOURDEAUX. *Bull. sci. pharmacol.* 28, 145-55 (1921).—A report of a study of the oxidizing action of pure $Na_2S_2O_8$ on org. and inorg. solns. in the presence of a catalyzer (Ag), in alk. solns. with and without catalyzers (Ag, Cu), and successive oxidation in alk. solns. without catalyzer followed by reaction in acid soln. plus catalyzer. Ag particles in the presence of $Na_2S_2O_8$ in acid soln. are changed to a compd. of unknown compn. which D. has called Ag peroxide, and which in the presence of oxidizable substances is reduced immediately, and then reoxidizing continues the process of O_2 transfer until the reaction is complete. The termination of the reaction is denoted by a brown coloration of the soln., which is attributed to the permanent production of the Ag peroxide. This compd. however, continues its action as long as there is $Na_2S_2O_8$ in the soln. Metallic Ag alone is capable of acting as a catalyzer. The pptn. of Ag as AgCl when Cl is present in the soln. decreases the available catalyzing capacity, since the reaction is more rapid the more free Ag particles are present. In non-catalyzed alk. solns. the stability of the persulfate with reference to temp. changes is much greater than in the previous case and the reaction is complete when the soln. is colorless, when it gives no charring on evapn. and ashing, and no $KMnO_4$ reduction. Successive oxidation in non-catalyzed alk. soln. and Ag-catalyzed acid soln. is efficacious when complex compds., usually oxidizable with difficulty, are to be analyzed.

F. S. HAMMETT,

Trihalogen-methyl reactions. III. Use of the silver cathode in electrodeposition of copper. HOWARD W. DOUGHTY AND BENJAMIN FREEMAN. *J. Am. Chem. Soc.* 43, 700-4 (1921).—Pt electrodes can be replaced by Ag electrodes in the detn. of Cu by electrodeposition. The deposited Cu can be dissolved by an ammoniacal soln. of CCl_3COONH_4 , which leaves the Ag unattacked.

C. R. PARK

The volumetric determination of tin with potassium bromate. V. VELICH. *Chem. Listy* 15, 5-8, 38-41, 56-9 (1921).—The method as proposed by Fichter and Müller (*C. A.* 7, 2173) is more fully developed. The reduction of solns. of $SnCl_4$ in prepn. for titration is best conducted in a closed flask, provided with a Fresenius stopper, an inlet tube for the addition of liquids, and an inlet tube for the continuous passage of CO_2 . Al is the best reducing agent but in the detn. of Sn in alloys the presence of Ni during the reduction with Al aids materially. Metals which are pptd. during the Al reduction and which do not redissolve in HCl are removed by filtration through an asbestos mat under reduced pressure and in an atm. of CO_2 to prevent re-oxidation. It is best to use 0.2 *N* soln. of $KBrO_3$ (kept in an atm. of CO_2) for the titration. Various indicators belonging to the oxazines, thiazines and azines were tried. Capri blue (L) is the most suitable for this reaction. The above method gives satisfactory results for the detn. of Sn in relatively pure preps. and in alloys if these contain less than 0.3% of Fe.

JOHN M. KRNO

The detection of tinstone. WILHELM BILTZ. *Chem.-Ztg.* 45, 325 (1921).—If tinstone is moistened with acid and brought into contact with a strip of Zn the mineral takes on at metallic white appearance at the points of contact. B. points out that

this is caused by Sn and emphasizes the fact that not all samples of tinstone give the test.

S. G. SIMPSON

The colorimetric determination of iron in silicates with ammonium thiocyanate. JOSEF MATEJKA. *Chem. Listy* 15, 8-13(1921).—Thomson (*J. Chem. Soc.* 47, 493(1895)) and Proskauer made use of NH_4CNS for the colorimetric detn. of Fe. The procedure adopted by M. is as follows: Into a 20-cc. colorimetric tube place 1-2 cc. of standard Fe soln. (a soln. of $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ in 0.5% H_2SO_4 giving a concn. of 0.01 g. of Fe_2O_3 per l.), 8 cc. of a mixt. consisting of 1 part of 10% HNO_3 and 3 parts of 10% citric acid by vol., and then add 8 cc. of a 10% soln. of NH_4CNS . Dil. the mixt. to the 20-cc. mark. To two other tubes add the same solns. with the exception of the one containing the Fe and dil. to a vol. of about 19 cc. To one of these add drop by drop, until the intensity of coloration is the same as in the standard tube, the soln. of the sample to be tested, obtained by dissolving its pyrosulfate fusion in 0.5% H_2SO_4 and dilg. to 250 cc. Then make a second detn., taking care to have the final vol. in the colorimetric tube exactly 20 cc. The following precautions are to be observed. The thiocyanate must be added in excess and its concn. must be the same in both tubes (cf. Tatlock, *Z. anal. Chem.* 28, 706(1889)). K_2SO_4 and KHSO_4 cause a reduction of the intensity and change the color from pink to orange. NaCl , MgCl_2 , CaCl_2 , TiCl_4 and Al salts have a detrimental effect. The interference of all these salts is inappreciable if the soln. to be tested is sufficiently acidified with HNO_3 and citric acids before the addition of thiocyanate. The concn. of the Fe in the soln. in the tube should be between 0.00001 and 0.00002 g. Fe_2O_3 . The reagents should be absolutely free from Fe. M. has found the above method as satisfactory as the ether method of Lunge (*Z. angew. Chem.* 1886, 3).

JOHN M. KRNO

The gas-volumetric method for determining carbon in steel and iron. HERMANN BURKARDT. *Chem.-Ztg.* 45, 342(1921).—Brief notes on the manipulation of app.

S. G. SIMPSON

The cyanometric assay of nickel. G. H. STANLEY. *J. S. African Assoc. Anal. Chem.* 10, 12(1921).—When Fe is pptd. by NH_4OH some Ni is always carried down but if the pptn. takes place in the presence of excess KCN the $\text{K}_2\text{Ni}(\text{CN})_4$ is not absorbed. The excess KCN can then be detd. in an aliquot part of the filtrate. To det. Ni in steels, dissolve 2 g. in HCl , oxidize with HNO_3 , add 3 or 4 cc. of dil. H_2SO_4 and nearly neutralize with ammonia. Add a measured vol. of standard KCN soln. and then enough NH_4OH to ppt. the Fe. Make up to 500 cc., mix and pipet off 50 or 100 cc. of the filtrate. Add a few drops of 10% KI soln. and titrate with standard AgNO_3 soln.

W. T. HALL

Comparison of different methods of estimating sulfur in steel. T. E. ROONEY. *Iron and Steel Inst.*, May 1921 advance copy, 13 pp.—Many analyses were made to det. whether it is necessary to anneal the drillings used in the volumetric detns. Correct results were obtained by the usual iodometric method in the case of all the ordinary steels tested except when the drillings had rusted and the only effect of the annealing was to reduce the oxide when filter paper was used to wrap the sample. Some samples of Cr-Ni steel gave decidedly low results by the iodometric method but there was no appreciable improvement by annealing in various ways and the cause of the low results could not be ascertained.

W. T. HALL

Preparation of zirconia from Brazilian ore and a new method of determination (ROSSITER, SANDERS) 18. Behavior of copper in molybdenum ores (BONARDI, SHAPIRO) 9.

RUDISÜLE, A.; *Nachweis, Bestimmung und Trennung der chemischen Elemente.* Vol. V. Bern: Akad. Buchhandlung vorm. Max Drechsel. M 70. For review see *Chem. Tech. Ztg.* 37, 64(1919).

8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND WALTER F. HUNT

Calculation of refractive index in random sections of minerals. LEO A. COTTON AND MARY M. PEART. *J. Proc. Roy. Soc. N. S. Wales* 54, 177-87(1920).—The principle of the method is developed mathematically by C., and applied by P. to the calcn. of the n_s of various feldspars.

JEROME ALEXANDER

Arsenopyrite twins from New Mexico. D. D. SMYTHE. Cornell Univ. *Am. Mineral.* 6, 85-6(1921).—The crystals are briefly described and figured. The angles are somewhat abnormal. Variable amts. of Cu were found to be present, evidently due to admixed chalcopyrite. Pyrite was also visibly admixed, and as sepn. of these impurities was impracticable no analysis was made.

E. T. W.

The identity of "collbranite" with ludwigite. EARL V. SHANNON. U. S. Nat. Mus. *Am. Mineral.* 6, 86-8(1921).—A black mineral from the Suan concession, Korea, has been supposed to be ilvaite, also a ferriiferous pyroxene, assigned the name collbranite (Higgins, C. A. 12, 1276). Material agreeing with these in properties has been now analyzed, giving: SiO_2 0.40, Fe_2O_3 32.49, Al_2O_3 2.32, B_2O_3 16.80, FeO 10.40, CaO 1.88, MnO 0.36, MgO 34.54, H_2O 1.42, sum 100.59%. This shows it to be actually ludwigite.

E. T. W.

Chemical investigation of Japanese minerals containing rarer elements. I. Analyses of naegite, fergusonite, and monazite, from Naegi, Mino Province. YUJI SHIBATA AND KENJIRO KIMURA. *J. Chem. Soc. Japan* 42, 1-16(1921).—All the minerals used were carefully selected and identified. Detailed descriptions of the minerals and method of chem. analysis are given. The results of analysis are: (1) Fergusonite, sp. gr. 4.4-2: SiO_2 2.83 (Cb, Ta) $_2\text{O}_5$ 43.77, TiO_2 1.65, ThO_2 2.91, CeO_2 1.26, Nd_2O_3 and others 1.99, Y_2O_3 and others 37.64, UO_2 0.85, Fe_2O_3 3.97, Al_2O_3 0.2, CaO 1.16, MgO trace, loss below 110° 0.24, and above 110° 1.85%. The spectrum analysis for rare elements shows that in Ce group, besides Ce, Nd is the greatest, then Sa and La, and that in Y group Y, Dy, Er, Yb are greater, then Gd. Similar results were obtained from 4 different samples. Presence of SiO_2 in Japanese fergusonite is the point different from the foreign minerals. Ti is always present in the Japanese mineral. (2) Naegite, d. 4.1, was analyzed in 6 different samples, the results of the last two being: ZrO_2 53.03, 51.24%; SiO_2 29.55, 30.48%; (Cb, Ta) $_2\text{O}_5$ 1.12, 4.34%; ThO_2 and others 9.53, 8.40%; UO_2 2.69, 2.30%; Fe_2O_3 1.42, 1.38%; ign. 2.77% (0.72 below 110° and 2.05 above 110°), 2.58%. The spectra of rare elements show that Ce and Nd give the largest no. of bands in Ce group, than La, Pr and Sa. In Y group, Y, Dy, Er, Yb, next Gd, Th, Ho. According to S. and K. naegite is an isomorphous mixture of ZrO_2 , SiO_2 , ThO_2 , and UO_2 , containing mixed crystals of rare earth salts, and silicates. (3) Monazite contains SiO_2 10.81, ThO_2 8.52, Ce_2O_3 19.44, Nd_2O_3 19.70, Dy_2O_3 and others 3.54, P_2O_5 20.33%, TiO_2 , Cb_2O_5 , Ta_2O_5 , ZrO_2 trace. Al_2O_3 , Fe_2O_3 , CaO , MgO were not detd. Rare element spectra show, in Ce group, that Ce, Nd are greatest, then La, Sa and Pr in order. In Y group, Dy is greatest, then Yb, Gd, Er next, then come Eu, Ho, and Tm, Lu the least. The sample contained 70% of pure monazite. S. T.

The geology of the gold occurrences of Victoria, Australia. N. R. JUNNER. *Econ. Geol.* 16, 79-124(1921).—All workable deposits consist of free milling Au in quartz, with considerable carbonates. The ores fall into 6 classes according to the typical associates: (1) Stihnite, (2) sulfarsenides and sulfantimonides, (3) pyrite and arsenopyrite, (4) galena, sphalerite, and pyrite, (5) pyrrhotite, (6) high temp. minerals. The Au was introduced into the country rock by alk. solns. of carbonates and sulfides, which were reduced by carbonaceous material from the sedimentary rocks. Secondary enrichment by the downward migration of meteoric waters gives a maximum concn. at 200 ft.

CHESTER B. SLAWSON

The limestone ores of Manhattan, Nevada. HENRY G. FERGUSON. *Econ. Geol.* 16, 1-36(1921).—These ores are replacement deposits in a thin bed of limestone. Replacement occurred with intrusion of an adjacent andesite porphyry and is limited to a well defined area in which mineralization has been controlled by numerous fault planes. Free Au is found associated with pyrite, leuconite, fluorite, and adularia but the most productive ores are auriferous arsenopyrite and secondary realgar and orpiment. In some instances realgar is so abundant that it has been smelted as an ore of As.

CHESTER B. SLAWSON

The magnetite ores of North Carolina; their origin. W. S. BAYLEY. *Econ. Geol.* 16, 142-53(1921).—These ores are of 2 essentially different types: (1) Titaniferous with 4.5%-41% TiO_2 and small quantities of Cr_2O_3 and only traces of MnO_2 ; and (2) non-titaniferous with less than 0.25% TiO_2 and considerable MnO_2 . A common origin has been ascribed to these ores but B. finds that the titaniferous ores are associated with other decomn. products from schistose peridotites and were formed subsequent to the general deformation of the region. The non-titaniferous ores antedate the titaniferous ores and are the result of a series of magnetite and pyroxene intrusions that have been subjected to considerable deformation.

CHESTER B. SLAWSON

Iceland spar. OLIVER BOWLES. *Bur. Mines, Repts. of Investigations*, 1920, (Ser. No. 2238), 6 pp.—A general description of Iceland spar, its properties, uses and sources of supply.

JEROME ALEXANDER

Some criticisms of Iddings' classification of igneous rocks. VICTOR HACKMAN. *Bull. Comm. Geol. Finlande* 53 (1920).—H. criticizes div. 2 and 3 of I.'s classification. He makes no objection to ratios quartz: feldspar and alkali feldspar: soda lime feldspar, but would change certain parts of I.'s terminology which places too great limitation on granite. This causes widening of the breach between established geological terminology and special petrographical nomenclature. Granites are not limited to the small minority designated, but are distributed over all parts of div. 2, chiefly between quartz-monzonite and quartz-diorite. Monzonite.—W. C. Brögger's work classifies rocks from Monzoni (Tyrol) as acid and basic and excludes latter from name monzonite. H. presents table showing monzonite classified between syenite and diorite. The position of quartz monzonite is also shown. He and I. made same use of terms monzonite and quartz-monzonite. I. fixed $<1/2, >1/2$ for monzonite; B. calcd. mode of monzonite from Monzoni, finding same ratio. J. Romberg describes several of these characterized by $<1/2, >1/2$; $<1/2, >1/2$; and $<1/2$. Occurrences of rocks similar to these are general throughout the world. This shows that monzonite: occurs universally with alkali rocks; carries to certain extent ingredients characteristic of same (Na-orthoclase, barkevikite, arfvedsonite); is characterized by quite basic plagioclase; exhibits direct geologic transition to basic members of alkali rocks. Its position is shown to be between syenite and theralite. Monzonite is therefore a special variety between syenite and diorite and is characterized by distinct approach to basic members of alkali rocks. Quartz-monzonite.—J. Romberg describes a prototype from Tyrol showing quartz: feldspar 5.65 $<1/2$. A monzonite aplite from same source contained 17 and an American one 19-25% quartz. Even if assumed that quartz-monzonites as a whole contain sufficient quartz for classification in div. 2 it is incorrect to use this term, originally designating a rare rock, as a name for rocks as common as granites; and an unsuitable name in this division may cause more trouble than in others. Granodiorite as used by I. has become better known than quartz-monzonite in petrographic literature but granodioritic granite has persisted even here. Lindgren's analyses show alkali feldspar: Na-Ca-feldspar, $<1/2, >1/2$ which would exclude California granodiorites from classification by I. as such and make them orthoclase-quartz-diorites. Agreement of av. of chem. analyses of 47 granodiorites (H. S. Washington) with av. of Lindgren's analyses indicates that the above ratio would hold for a majority of the

47. I.'s values for granodiorites would include some granites, especially oligoclase granites. His system is misleading when applied to Finnish granites of which 5 can scarcely be distinguished from granodiorite; another would be quartz-monzonite; and a seventh orthoclase quartz-diorite. The av. of chem. analyses of 8 Finnish and Swedish granites shows important differences when compared with chem. analyses of granodiorites mentioned above, especially in SiO_2 (higher in granites), CaO and MgO (lower), and in alkali ratio (K_2O higher than Na_2O). *Granodiorite* should replace *orthoclase quartz-diorite* and *quartz-monzonite* and *monzonite* should be eliminated. A change in terminology is shown by a table in which are shown I.'s classification and the suggested scheme.

G. R. J.

Petrography and mineralogy of limestone deposits of Parainen (Pargas). AARNE LAITAKARI. *Bull. Comm. Geol. Finlande* 54, 114 pp. (1920).—The rock of this region is chiefly granite and gneiss but limestone occurs in long narrow strata running east and west. This is a small part of widely scattered deposits formerly existing and which have been buried by folding. It is white, coarsely cryst., and dolomite-free. Ca-gneiss lies immediately south of the limestone and consists of plagioclase, diopside and microcline. Amphibolite is in turn south of the Ca-gneiss. These three rock strata are intruded by many granitic and basic veins which are described in detail, especially pegmatite and diabase. Folded and faulted bodies of gneiss occur at times, within the limestone veins. Position and nature of intruded minerals indicate origin in impure limestone sediment. Most of the limestone is a wollastonite complex and contains pyrrhotite instead of pyrite. Other minerals occurring in the limestone are: graphite, galenite, chalcopyrite, löllingite, fluorite, quartz, ilmenite, spinel, microcline, plagioclase, diopside, wollastonite, tremolite, actinolite, pargasite, scapolite, vesuvianite, orthite, chondrodite, tourmaline, phlogopite, clintonite, talc, titanite, and apatite. These may constitute 20% of the rock. A deposit containing pure quartz and limestone and one of wollastonite and limestone are described. The probable origin of these is discussed. Ca-gneisses of 3 types are described and measurements of mineral constituents (microcline, plagioclase, diopside, wollastonite, titanite, and calcite) given. Amphibolite occurs both stratified and intrusive, these being petrographically similar. Quartz bearing amphibolite containing plagioclase, quartz, diopside, and biotite is mentioned. Small deposits of grossularite and diopside vary from equal parts to almost pure diopside. Mineralogical descriptions of minerals accompanying the limestone are given. Scapolite shows some new faces. Frequent forms are (100) and (110). Others (310), (331), (301), (611), (211), (112), (212), and (332) are certain. Most common combination is (100), (110), (111), (001), (311); nine crystals are described. Minerals surrounding the limestone form with it a variety of contact zones. These include one or more minerals, especially grossularite-vesuvianite, chondrodite-spinel, chondrodite-pargasite, apatite-fluorite, plagioclase-diopside, and scapolite-diopside. One seam shows well preserved pseudomorphs of garnet in dodecahedral crystals. The seams vary in thickness from a few mm. to several cm. One contact with a granite vein shows graphite deposited and this in turn covered with symplectic plagioclase-diopside. A three-part seam is described. A sketch and description of the contacts among limestone, granite, amphibolite, plagioclase, and grossularite are given. "Scale forms" of minerals, up to 0.5 cm. thick, frequently overlie a part or the whole of an individual of another mineral. Pargasite, diopside, vesuvianite, fluorite, phlogopite, and chondrodite scales are described. Scales were formed by reaction between host mineral and a substance added externally; also by surface crystn. Paragenesis of minerals common to this territory is shown by a table. The constituents of the original limestone sediment do not account for the minerals now present. Extraneous material must have been added, especially Cl, F, S, P, and Ti, also some As. They were probably added in both gas and liquid forms. The Mg con-

tent is lower than would be expected from the minerals assumed to have been added. Metamorphism was regional and probably caused by granitization. A granite eruption and palinogenetic formation of gneiss veins developed conditions for completion of metamorphism. Evidence of stratification of the limestone preclude the assumption of metamorphism in condition of magna. Bibliography, 10 microphotographs and 40 illustrations are included.

G. R. J.

The origin of the alkali igneous rocks. J. W. EVANS. *Rept. Brit. Assoc. Advancement of Science* 1920, 354.—These rocks, distinguished by unusually high proportion of alkalis, relatively to alumina and lime, occur mainly where the crust of the earth is thick, the heat-gradient low, and there has been no folding since remote times. Magmas appear to have reached the surface by fault fissures from great depths where high pressures are associated with comparatively low temperatures. Crystn. proceeding under such circumstances, there would be an early formation of minerals with small mol. vols., garnets, kyanite, epidote, and zoisite, minerals rich in lime and alumina. Zoisite may be regarded as the high-pressure representative of anorthite, but there is no corresponding representative of albite or orthoclase. Consequently one should expect a residual magma exceptionally rich in alkalis which would furnish the material necessary for formation of alkali rocks.

E. H.

Oil field brines. C. W. WASHBURN. *Mining & Met.* 1920, No. 164, 27.—W. discusses the paper of Mills and Wells (*C. A.* 13, 1573) on a chem. study of waters associated with oil. M. and W. are credited with presenting good arguments for the hypothesis that the concn. of deep brines was produced by evapn. of sea water in rock pores induced by migrating gas escaping upwards. The concn. of H_2O in sands, nature of salt (cores) and the origin of salt core caps and the hard caps of oil sands are mentioned.

J. A. RUMSEY.

American enterprise makes "rare earth" a nuisance (PRITCHARD) 18. Detection of tinstone (BILTZ) 7. Underground conditions in oil fields (AMBROSE) 22.

GROSSMANN AND NEUBURGER: Die synthetischen Edelsteine, ihre Geschichte, Herstellung und Eigenschaften. 2nd Ed. Berlin: M. Krayn. 72 pp. M 4. For review see *Chem. Weekblad* 18, 210(1921).

9—METALLURGY AND METALLOGRAPHY

D. J. DEMOREST, ROBERT S. WILLIAMS

The dressing of ores by the flotation process. BRUNO SIMMERSDACH. *Chem. Ztg.* 45, 357-60, 383-5(1921).—A general elementary description of the flotation process.

R. S. DEAN

Cadmium in 1920. C. E. SIEBENTHAL AND A. STOLL. U. S. Geol. Survey, *Mineral Resources of U. S.*, 1920, Part I, 1-6 (preprint No. 1, published May 12, 1921). E. H.

Determination of the quantity of gas evolved during zinc distillation. O. MÜHLHAUSER. *Metall u. Erz* 17, 415-20(1920).—Tables and graphs are given showing the temp. and pressure in the retort and the volume and N content of the gases evolved during the progress of the distn. of Zn from ZnO (roasted blende) and anthracite. The rate of evolution of the gases is greatest at the commencement of the operation, when the temp. of the retort has reached 1000° , and falls rapidly during the next two hrs., after which it again rises with the rise in temp. to a second max., which, however, is much lower than the first. The rate then gradually decreases until the end of the distn. The N content of the gases is at a max. at the beginning then slowly decreases until the middle of the distn., after which it again rises to a max. at the end of the operation.

J. S. C. I.

Influence of foreign gases on the formation of zinc dust during the condensation of zinc vapors. O. RAVNER. *Tekn. Ukeblad* 67, 404-8, 418-20(1920).—In order to minimize the deposition of Zn dust during condensation of Zn vapors the following points should be observed. The temp. of the receiver should be a little above the m. p. of Zn and the distance between the retort and the receiver should be as small as possible, as the longer the time that elapses before the vapors condense the greater is the amt. of CO formed by decompn. of the CO₂ present. This leads to the formation of films of ZnO on the droplets of condensed Zn, and prevents them from running together. Similar effects are obtained if nitride is formed, either from N in the coke or from leakage of air into the retort; or if the ore or fuel contains S, ZnS being slightly volatile. Traces of moisture in the charge lead to the formation of dust in the pipes, and, in general, the more the Zn vapors are diluted with foreign gases the greater is the dust deposit. Chem. and screen analyses are given of typical coarse and fine Zn dust. In order to obtain a metallic regulus from the dust the best process is to melt it with a mixt. of equal parts of Zn and Ca chlorides; in one case a yield of 91% of the total metal present was obtained in this way. J. S. C. I.

Experimental furnace for the preparation of zinc and the determination of the various products. O. MÜLLHAUSEN. *Metall u. Erz* 17, 487-94(1920).—Minute details accompanied by the necessary drawings are given of a small exptl. plant for the distn. of Zn and the collection and treatment of the various products. The plant consists of a gas producer, retort, and combustion chamber, a condenser for Zn, and scrubbing towers to remove the fume from the issuing gases. Exact instructions are given for working the furnace, together with three examples showing the results obtained from charges of ore, crude Zn (90.6% Zn), and Zn dust. J. S. C. I.

The Almaden quicksilver mine in Spain. H. H. GOULD. *Mining Sci. Press* 122, 567-9(1921).—The ore and process of treatment are described. A. H. HELLER

The Arizona Silver Mines mill. F. BORZYNSKI. *Mining Sci. Press* 122, 717-8(1921).—First attempts at cyaniding proved unsuccessful, and straight flotation concn. gave a concentrate too low in grade to be marketable at a profit. The present mill equipped with K. and K. flotation machines, vanners and a cyanide plant was started in October 1921. Flotation proved to be superfluous, the vanner concentrate assaying 350-500 oz. Ag/ton. The ore consists of a mixt. of SO₂ and CaCO₃ with small amts. of pyrite, stibnite and scheelite. The valuable minerals are argentite and bromide and chloride of silver. Over 60% of the run-of-mine ore passes 4 mesh, the remainder is crushed sufficiently to be fed to the five 1050-lb. stamps, from which it passes to a 48-in. Aikens classifier in closed circuit with a 4 × 9-ft. tube mill. After passing over vanners the pulp goes to the cyanide plant consisting of Dorr thickeners and agitators. Strength of soln. is maintained at 4 lb. cyanide per ton. The pulp is dewatered in a 12 × 8-ft. Oliver filter. Twenty tons are treated daily; the cyanide consumption is 2.7 lb., lime 1.5 lb. and lead acetate 0.3 lb. A. H. HELLER.

Recent metallurgical practice on the Rand. ANON. *Eng. Mining J.* 111, 623-4(1921).—With the exception of the adoption of direct leaching in the sand collectors without transfer, there has been no notable change in metallurgical practice in the gold fields of the Rand in the last 10 years. H. C. PARISH

The Alaska Juneau Mill. V. C. CLAUSON. *Mining Sci. Press* 122, 623-34(1921).—A record of events leading up to and influencing its design and construction. The early history of the district and its mills is given with a table of operating costs of the Alaska Juneau 30 stamp mill from 1901 to 12. A description of the flow sheet of the Alaska Gold Mill is given. A. H. HELLER

Treatment of a gold ore. ROBERT PAULIN. *Chem.-Ztg.* 45, 285-287(1921).—The ore treated was taken from old heaps at Krasna Hora in Bohemia. It contained native gold in quartz, stibnite iron pyrites and arsenic ore in small quantities. The

gold content was 2-8 g. per t. and the antimony 2-4%. A concn. works, supposedly to handle 70 t. of ore per 24 hrs. including one Ferraris shaking hearth and two amalgamation tables were at hand. Following the ordinary process of concn. and sepn., the ore is passed on to the Ferraris hearth where a heavier concentrate containing most of the gold and a middle concentrate carrying the stibnite and the pyrites are obtained. Then the gold concentrate is passed into the amalgamation tables, of which a description is given. A copper plate is used, preferably electroplated with silver, into which the mercury is rubbed, or silver-amalgam previously prepd. may be applied directly. Care had to be taken that (1) the amalgamation plates be kept very pure, (2) the ore have the right size so as not to slime too much and still allow most of the gold to amalgamate and (3) sufficient time be given the gold to amalgamate on the plates. Tests with 0.18 kg. gold concentrate from the hearth contg. 96.38 g. Au showed after 14 days' treatment that only 12.15 % of the gold had been recovered in the amalgam, the rest having been attached to or surrounded by quartz pieces. A trap for the lost amalgam consisting of a crock with an overflow was attached to the tables and emptied every 14 days. From the overflow some gold was recovered by gravitation and this together with the contents of the crock was amalgamated in a special drum, enough mercury being present to keep the amalgam formed liquid. The mercury is pressed out of the amalgam, which is then heated in a furnace to dist. off the rest of the mercury. The gold residuc is remelted with soda. G. N. KIRSEBOM

Government armor-plate factory. SONEY G. KOON. *Iron Age* 107, 1365-71 (1921).—The Naval Ordnance plant at South Charleston, W. Va., is described with illustrations. E. H.

Utilization of waste of steel works and foundries, particularly the recovery of iron from sweepings. HUBERT HERMANUS. *Met italiana* 12, 182-92 (1920).—H. describes a mechanical method of carrying out the purpose stated in the title. Sketches of the devices used are given. ROBERT S. POSMONTIER

Blast-furnace and cupola slags. Their composition and graphic methods for determining their constitution. J. E. FLETCHER. *Trans. Iron & Steel Inst.* Advance copy, No. 3 (1921).—F. gives a series of graphs which aim to provide a clearer view of the slag soln. theory and of its practical utility. Though the mineralogical character of the cold slags is useful in slag research there is danger of assuming that the many cryst. transition products in the cooled slags were present in the molten mass. Also in *Engineering* 111, 760-2 (1921). H. C. PARISH

Modern metallurgical laboratories. A visit to the steel foundries of Essen. ANON. *Age de fer; Met. italiana* 12, 335-7 (1920).—Descriptive. ROBERT S. POSMONTIER

Cleaning of blast-furnace gas. S. H. FOWLES. *Trans. Iron & Steel Inst.* Advance copy, No. 4 (1921).—Waste gas and heat have been used as efficiently in England as on the continent and in America. There is a productive field for potash recovery from the crude gas dust. If the gases be well cleaned and efficiently used it is possible to produce speeding up works, and undertaking much of the work now done by coal-fired boilers. It would be possible to supersede the old steam-driven blowing-engines and render blowing by large and efficient gas-driven engines easy. The elec. equipment of large works in England 3-4 million h. p. from waste blast-furnace gases, reducing large coal bills, can be developed on a larger scale and at lower cost than was ever anticipated. 40% of crude gas has been consumed in hot stoves and 60% in boilers. To use the gas under boilers, whether from coke ovens or blast furnaces, is a wasteful practice. The internal-combustion engine is considered the most efficient method of utilizing this gas. Gas-cleaning plants are of 3 types: (1) wet process, (2) dry process and (3) electrostatic process. It is necessary to dry the gas from the wet process for engine consumption. This means higher maintenance costs on the engines. The gas from the electrostatic process after cleaning contains all of its sensible heat. It is, however, not

sufficiently clean for engine consumption and must be passed through another type of cleaner. The power and capital costs are very high. The dry process produces a gas possessing all advantages required by a gas engine and is free from the objections of the other types. It is moderate in first cost, economical to maintain, easy to operate and flexible in respect of furnace operation. It produces a clean dry gas of reasonable temp. for stoves and industrial heating. Furthermore a continuous gas supply is maintained when the fans are accidentally stopped. H. C. PARISH

Behavior of copper in molybdenum ores. J. P. BONARDI AND MAX SHAPIRO. *Chem. Met. Eng.* **24**, 847-50(1921).—Copper minerals occur in some Mo ores and are not wholly eliminated in concg. A max. limit of 0.5% Cu is desirable if the concentrate is to be used for Fe-Mo manuf. Larger amts. of Cu do not interfere with the use of Mo concentrates for making Mo compds. by fusion with alkali. Gravity concn. with vanners effects a fair sepn. of Mo and Cu minerals. Oil flotation concentrates sulfides of Mo and Cu together. Differential flotation is promising. Electrostatic methods fail to differentiate. Chalcopyrite and pyrite can be sepd. from MoS by a roast which renders them magnetic, followed by magnetic treatment. If the ratio of Cu to Mo is not too large, it is possible to make concentrates with less than 0.5% Cu. In the *analysis of Mo ores and concentrates for Cu* the direct iodide method fails because Mo also liberates I. It is not possible to ppt. Cu by Al when Mo is present and granulated Zn, while efficient, produces finely divided Cu. Pptn. by NaOH if boiling is sufficient seps. Cu from Mo but is tedious. Modified electrolytic methods for Cu in the presence of Mo are inadvisable. The thiocyanate method, somewhat modified, gives an accurate sepn. Heat an amt. of finely pulverized ore contg. not over 0.20 g. of Cu with 15 cc. of HNO₃ in a 250-cc. beaker until brown fumes cease. Add 10 cc. HCl and boil nearly to dryness. Cool, dil., add 15 cc. of 1:1 H₂SO₄ and H₂O enough to dissolve the salts. Evap. to strong fumes. Cool, dil. slightly, heat, filter and wash the residue. Make the soln., now measuring about 250 cc., alk. with NH₄OH and then acid, leaving 2 cc. of free 1:1 H₂SO₄. Next add 3 g. of anhydrous Na₂SO₄ to reduce the Cu. Redissolve any ppt. with a little dil. acid. Heat nearly to boiling and add 10 to 25 cc. of a soln. of NH₄CNS or KCNS contg. 1 g. in 5 cc. Heat until coagulated well. Filter and wash well with hot water. Dissolve in 10 cc. warm 1:1 HNO₃. Evap. practically dry, cool and add 50 cc. warm water and 2 or 3 drops of HNO₃, if necessary, then 5 to 10 drops of NH₄OH or enough to make a deep blue. Next add 5 to 10 cc. AcOH and 5 to 6 cc. of KI soln. (50 g. in 100 cc.) 2 Cu liberates 2 I. N oxides, ferric ions and tervalent As and Sb must be absent. Br water converts As and Sb in the HNO₃ soln. to quiescent forms. Excess Br is expelled by evapn. Cu detns. by this method are exact. J. O. H.

Microstructure of chromium steels. ANON. *Chem. Met. Eng.* **24**, 703-6(1921).—A brief review is given of the literature, especially the work of Murakami, with structural diagrams, micrographs and an explanation of the changes in transformation temp. following various reheatings; also magnetic analysis. "...the self hardening of a Cr steel is related to the lowering or complete suppression of the Ar transformation, the hardness being caused by the solid soln. of Cr₂C₃ in iron which has already dissolved chromium. Steels having the normal transformation point show troostitic or pearlitic structures, while those having the lowered transformation are martensitic. Where this transformation is completely suppressed the steels are austenitic."

JEROME ALEXANDER

Structure of tungsten steels. ANON. *Chem. Met. Eng.* **24**, 745-8(1921).—Mainly a review of the work of Honda and Murakami, including structural diagram of tungsten steels, magnetic analysis, micrographs, etc. Reactions between iron tungstide and carbides of iron and of tungsten, are responsible for great complexity. The main conclusions are: (1) The presence of two carbides, Fe₃C and WC, in magnet steel as found by Arnold and Read is very probable. They may exist separately or as a double

carbide, depending upon heat treatment; *i. e.* they exist as double carbide after beating once to 800–900° and slowly cooling; (II) above Ac, this double carbide decomposes, both its components remaining dissolved in austenite up to about 900°. On further beating WC decomposes into W and C, which remain in solid soln., while the W dissolved in the austenite forms Fe₂W. The dissociation is practically complete at 1100°; (III). By normal cooling from above 1100° the A₁, A₂, and A₃ transformations are lowered to a temp. below that of the normal A point by the retarding effect of the Fe₂W dissolved in the austenite. At about 500° the retarded A₃ transformation begins to take place and with it the ferrite (with dissolved Fe₂W) separates from the solid soln. This continues for about 50° until the concn. of the remaining mother soln. reaches such a value that the A transformation takes place at this temp. JEROME ALEXANDER

Electric resistance of nickel steels. A. PORTEVIN. *Compt. rend.* 172, 445-7 (1921).—Duplicate series of Ni steels were given different heat treatments. One set of samples was heated to 1000° and cooled during a period of 4-5 hrs. The other set of samples was heated to 1300° and cooled during 3 days. In most cases the sp. resistance was the same for two samples of the same compn., but for certain steels contg. 0.3-0.8% C and 7-15% Ni the difference was as great as 40% (based on the sample of smaller resistance). The change in the resistance is closely connected with the presence of martensite. C. R. PARK

Strains in gray cast iron of different grades. O. BANSE. *Stahl u. Eisen* 39, 313-6, 436-41, 596-600 (1919).—The effect of the grade of the Fe and the moisture content of the mold on the appearance of strains in square or round frames of gray cast Fe with thin crossbars was detd. The strains produced diminish as the Si content increases, and are smaller in castings produced in dry molds than in those made in molds of "green" sand. J. S. C. I.

Progress made in Germany during the war in the field of heavy metallic alloys. GINO GALLO. *Giorn. chim. ind. applicata* 2, 562-5 (1920).—Review. R. S. P.

Use of hydrogen peroxide in metal tinting. H. KRAUSE. *Metall.* 1920, 229-30.—H₂O₂ is of no use for tinting smooth polished metal surfaces, but for unpolished surfaces, especially those that have been colored by other reagents, *e. g.*, a strong soln. of KClO₃ contg. NH₄NO₃, or for darker tints, KMnO₄ concd. solns. of H₂O₂ (perhydrol) are useful for toning down the colors, for which purpose the reagent is applied with a fine brush and develops generally a bright brown color. J. S. C. I.

Practical means of preventing corrosion of iron and steel where not exposed directly to the atmosphere (SPELLER) 14. The crystallization of metals by galvanic precipitation (ATEN, BOERLAGE) 2. Stannous chloride [refining tin] (Brit. pat. 159,659) 18.

GEE, GEORGE E.: **Recovering precious metals from waste liquid residues.** London: E. & F. N. Spon. 380 pp. 16s. net.

HOYT, SAMUEL L.: **Metallography. Part II. The metals and common alloys.** New York: McGraw-Hill Book Co. 462 pp. \$5. Cf. *C. A.* 14, 1656.

MARCON, E.: **Notions élémentaires sur la métallurgie et le travail des métaux ferreux.** 2nd Ed. Paris: Librairie Delagrave. 204 pp. 5 fr. 75 + 30%. For review see *Rev. Electrochim.* 15, 51 (1921).

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Metal statistics, 1921. New York: American Metal Market. 560 pp. \$1. For review see *Eng. Mining J.* 111, 715 (1921).

WCST, FRITZ: **Mitteilungen aus dem Kaiser-Wilhelm-Institut für Eisenforschung**

zu Düsseldorf. Vol. 1. Düsseldorf: Verlag Stahl Eisen. 120 pp. M. 60, bound M. 70
For review see *Z. angew. Chem.* 34, Aufsatzteil, 133(1921).

Extracting copper, gold and silver from ores. H. FOERSTERLING, ET AL. Can. 210,024, Apr. 5, 1921. The ores are treated with a cyanide soln. to ext. the metals and the soln. is reacted upon with a moving adherent film of an alkali metal amalgam capable of displacing the value in the soln. and of regenerating the original cyanide.

Recovering zinc from slags, ores, etc. F. C. W. TIMM. Can. 210,165, Apr. 5, 1921. Zn is removed as ZnO from zinciferous materials by smelting the material in contact with reducing substances and removing the ZnO from the slag by passing a current of heated gases downward through the charge.

Treating crude alkaline earth cyanide compounds containing calcium chloride. MATTHEW F. FAIRLIE and JAS. J. DENNY. Can. 210,502, Apr. 19, 1921. Crude Ca(CN)₂ contg. CaCl₂ is treated with Na₂CO₃ to ppt. the Ca and leave inert NaCl in soln. The accumulation of CaCl₂ in the solns. is thus prevented and its bad effects on the dissolving of precious metals avoided.

Treatment of slag from refining processes. GEORG LEDER. Ger. 322,797, July 10, 1918. The residuc of the charge flowing off from the furnace is directed upon an inclined plane, and the thin cake of slag and Fe which forms upon hardening on the plane is broken up into relatively small pieces by a liquid jet, while the slag is at the same time detached from the Fe. The iron-free slag running over from the pan is conducted upon a cast-iron cooling board located underneath or by the side of the pan. The slag which when the charge is tapped flows out over the lip of the pan and which contains relatively large amts. of Fe, is transferred by means of inclined troughs upon a cooling board, which consists of a frame and a bottom covered with iron plates or bricks and which allows the Fe flowing in to be deposited in a thin layer. After treatment with a liquid jet and after removal of the detached and broken-up slag, this layer of Fe is returned into the furnace, while the slag crumbling into small bits may be made ready to be loaded.

Blast-furnace stoves. JUSTUS M. RINGQUIST and THOMAS G. WRIGHTSON. Can. 209,101, Mar. 1, 1921. A blast-furnace stove is surrounded by an annular combustion chamber and a main for supplying gaseous fuel with connections between the main and the stove. The brickwork at the bottom of the combustion chamber is of undulatory form to provide dust-collecting spaces.

Blast roasting apparatus. GILBERT RIGG. Can. 211,038, Apr. 26, 1921. In blast roasting operations the permeability of the charge is maintained by periodically passing bars or teeth through the furnace grate to perforate the clinker.

Apparatus for concentrating phosphates and other minerals. CÉLESTIN POUPART. Ger. 324,571, Oct. 14, 1919. A washing-trough for the minerals to be concd. is formed of an endless belt or a piece of canvas, the edges of the belt or canvas being bent up by lateral rollers placed in oblique position. The app. extends through two water containers of different lengths. The level of the stratum of H₂O above the belt or canvas is capable of being regulated within certain limits.

Binding oxygen, sulfur and phosphorus from metal melts, more particularly from ingot iron and steel. DEUTSCH-LUXEMBURGISCHE BERGWERKS- UND HÜTTEN-AGT.-GES. and SIEGFRIED HILFERT. Ger., 322,988, Sept. 22, 1916. The metal melts are treated with Al₂C₃. The Al₂C₃ may be employed in mixture with such other carbides as to form by joint oxidation of the constituents a readily fusible slag. The Al₂C₃ combines with Fe to a certain extent, so that it becomes possible to bind the O contained in the Fe with much smaller amts., while the Al sulfides and phosphides formed rise in the bath by reason of their low sp. gr. and are then converted to slag. When the deoxidation is to be effected in absence of slag, hence in an ingot mold, the diffi-

cultly fusible Al_2O_3 is likely to remain suspended in the Fe and to cause fissures in the slag. In that case the Al_2C_3 is employed in mixt. with CaC_2 .

Refining shaped articles from manganese steels containing austenite. FRIEDR. SCHAFER. Ger. 321,035, Jan. 21, 1919. The shaped articles, made of a steel contg. a high % of addition of Mn, which tends to retard the sepn. of carbide, are first heated, for the purpose of refining, to above the critical temp. of cementite sepn. and then cooled by systematic arbitrary variation, effected in the course of the cooling process, of the heat capacities made to act per unit of time in accordance with a curve of time and temp. detd. according to the properties which the material is intended to possess. The shaped articles are made of a steel which contains, with about 1% of C, at least 12% of Mn.

Treating iron ores. R. STÖREN and R. JOHANSON. Brit. 159,380, Dec. 18, 1919. Magnetic Fe ores contg. pyrrhotite are heated in the presence of a reducing gas to a temp. of 400-500° in order to render the ferrous sulfide permanently non-magnetic, and the product is subjected to magnetic concn. H obtained from an electrolytic decompn. plant is preferably used. A suitable construction is specified.

Spongy iron. A. S. NORSK STAAL (ELEKTRISK-GAS REDUKTION). Norw. 31,324, Nov. 1, 1920. The spongy Fe, immediately after reduction of the ore is placed at full reduction temp. in H_2O which has either been thoroughly boiled or to which an alkali, *e. g.*, milk of lime has been added.

Spongy iron. ALF SINDING-LARSEN. Norw. 31,193, Oct. 4, 1920. In the reduction of Fe ore, concentrate or the like with H and CO the hot reduction zone is connected not only with a cooled zone lying at a higher level, in accordance with Norw. 29,475, but also with a receptacle located in an equally hot zone, CO_2 and steam being reduced by coal or coke.

Dense iron from spongy iron. ALF SINDING-LARSEN. Norw. 31,047, Sept. 20, 1920. The spongy Fe is subjected to mechanical treatment such as rubbing and grinding, and at the same time treated with CO at 100°. There then forms iron carbonyl, which is split by raising the temp.

Production of ingot iron and steel by deoxidation of baths rich in oxygen. DEUTSCH-LUXEMBURGISCHE BERGWERKS- UND HÜTTEN-AGT.-GES. and ADOLF KLINKENBERG. Ger. 322,752, Dec. 6, 1916. In the process set forth in 316,938 for the deoxidation of the ingot-iron and steel baths, the carbohydrates (cellulose, wood shavings, sawdust and the like) are now replaced by lignite and peat, while mineral coal has proved unsuitable for this purpose. The different action is probably due to the fact that peat and lignite on heating yield chiefly aliphatic distillates, while mineral coal forms aromatic products, which upon further decompn. cause considerable O deposits.

Iron and steel manufacture; furnaces. L. P. BASSET. Brit. 159,475, Feb. 24, 1921. See Can. 209,172 (*C. A.* 15, 1132).

Removal of the slag in the manufacture of ingot iron and ingot steel and of pre-refined iron in hearth furnaces. PAULINE THELNÉR MUCK. Ger. 322,798, May 13, 1915. Fresh crude Fe or other Fe is gradually added to the normal refining charge in the furnace during and after the refining process until the slag in the furnace has risen to such a level that it must run off. This takes place in tilting furnaces through the outlet with which they are provided, and in stationary furnaces through a lateral or bottom discharge such as may be fitted, *e. g.*, in the Hosch process, in the opening through which the pre-refined metal is intended to be poured back. Gradually the slag is entirely replaced by the Fe poured in. The Fe which alone fills the furnace, the surface being free from slag, will directly absorb an ample amt. of heat. The pre-refined Fe can be further treated to good advantage in a converter, where the duration of a charge is very short.

Spiegeleisen from pyrolusite. JULIAN HUBB. Ger. 322,610, Dec. 25, 1912. Pyro-

lusite, densely mixed with small coal or coke, is heated to redness in an auxiliary furnace, which is gradually filled up, the reduction being conducted at such high temp. that a slag forms which contains as far as possible only MnO. The slag is broken out from the bottom and transferred to a blast furnace. The O which abundantly seps. from the pyrolusite is of such violent action on the fuel that a very high temp. (white heat) is attained in a short time. The mixt., therefore, at once begins to melt, and a slag forms which contains practically only MnO. This slag is treated in the blast furnace like ordinary Fe ore and is claimed to cause very quiet working of the furnace.

Heat-treating steel. J. L. THORNE. Brit. 159,793, June 19, 1920. Steel articles, particularly cast-steel, are heated to a hardening temp. of about 2050–2350° F. and then immersed in a molten alk. bath such as molten NaOH at 1100–1250° F. until they acquire the temp. of the bath. They are then withdrawn and air-cooled to 600° F., whereupon they are immersed in a drawing bath preferably of the same compn. and same temp. or slightly higher than the first bath. After they have attained the temp. of the drawing bath, the articles are removed to acquire room temp. When treated by this method, the scale formed on the steel is readily removed by washing in H₂O, and when dried a black, rust-proof finish is given by applying oil to the surface of the article.

Iron alloys. SOC. ANON. DE COMMENTRY FOURCHAMBAULT ET DECAZEVILLE. Brit. 159,857, Nov. 5, 1920. Modifications of the Fe alloys described in 140,508 (C. A. 14, 2159) contain 50–80% of Ni, 8–25% of Cr, 1–5% of Mn, 0.3–1% of C, and either 0.5–8% of W or 0.2–5% of Mo; or W and Mo may be present together. One or more other metals may be present as follows: 0.2–1% of V, 0.1–0.2% of Ti, or 0.1–1% of V and Ti together, and up to 10% of Co.

Iron alloys. SOC. ANON. DE COMMENTRY FOURCHAMBAULT ET DECAZEVILLE. Brit. 159,858, Nov. 5, 1920. Modifications of the Fe alloys described in 140,509 (C. A. 14, 2159) contain 25–40% of Ni, 8–15% of Cr, 0.5–5% of Mn, and 0.3–1% of C. One or more other metals may be present as follows: 0.5–5% of W, 0.2–3% of Mo, or 0.2–5% of W and Mo together, 0.2–1% V, 0.1–0.2% Ti, or 0.1–1% of Ti and V, together, and up to 10% of Co.

Prevention of kish graphite formation in the manufacture of siliceous cast-iron alloys of high acid-resisting power. MASCHINENFABRIK ESSLINGEN. Ger. 321,879, Sept. 4, 1918. (Addition to 306,001). Steel or ingot Fe is produced by itself in a converter, a Siemens-Martin furnace or an elec. furnace, or steel or ingot-iron waste, or briquets produced from steel or ingot-iron shavings, are fused in any smelting app., but without crude Fe poor in C. On the other hand, the highly siliceous addition alloy is fused in a smelting app. which precludes enriching with C. The two melts thus obtained are mixed in any desired manner and poured into molds.

Iron or steel alloys. W. B. BALLANTINE. Brit. 159,280, Nov. 19, 1919. A process for making Fe or steel alloys consists in adding to a molten steel bath one or more of the alloying metals in the metallic form and also a quantity of a thermo-aluminic or like mixt. which contains one or more compds. of an alloying metal or metals. E. g., in making a steel alloy containing 17% of W, 3.5% of Cr and 0.5% of V, the W and V may be added as ground or granulated ferro-alloys in the form of a briquet or in a container, and the Cr as a constituent of a mixt. containing chromite and Al, which is inserted in the briquet or container, the complete mass being added to a molten steel bath. Chrome steel and steels containing Mn, Co, Ni or Mo may be similarly made, elements such as C or Si being added separately where necessary, preferably in a combined state. The Al in the mixt. may be replaced by other reducing agents, and the mixt. may also contain BaO, neutral substances such as CaO or MgO, or elements such as Mn or Ti for the absorption of gases. The thermo-aluminic mixt. may be arranged at the center or other part of the mass of the metallic element or elements, or the thermo-aluminic materials and the metals may be uniformly mixed. The ma-

terials for addition to the molten steel may be prepd. in the form of standard units, the thermo-aluminic mixt. being in one or more containers or blocks and the metallic element or elements in others, or the whole of the materials being in single units.

Production of cast-iron alloys poor in carbon. MASCHINENFABRIK ESSLINGEN. Ger. 322,236, Feb. 28, 1917. Wrought Fe or steel poor in C is fused in any suitable smelting app. An intermediate cast iron alloy is melted down in a cupola furnace without addition of materials poor in C. These two liquid alloys are then mixed in a known manner and finally poured into molds.

Ferrochromium. W. B. BALLANTINE. Brit. 159,568, Nov. 24, 1919. Low-carbon ferrochromium is made by subjecting crude ferrochromium to the action of an oxidizing blast in a converter. A thermoreduction mixt., preferably in the form of blocks or masses, and containing chromite or a pure Cr compd. or both, may be added to the molten alloy before or after tapping from the converter, for the purpose of adjusting the Cr content. The crude ferrochromium may be first prepd. by the reduction of chromite in an elec. furnace, steel scrap or the like forming part of the charge, or by a thermoreduction process in which a cheap reducing agent such as CaC_2 is used. In some cases, mild basic steel may be added to the crude material prior to treatment in the converter.

Alloys. ARMSTRONG, WHITWORTH & CO., LTD., C. J. G. MACGUCKIN AND O. SMALLEY. Brit. 159,537, Oct. 24, 1919. Cu alloys, especially suitable for turbine blades, contain 80-91% of Cu, 7-10% of Al, 1-6% of Mn and 1-4% of Fe. The alloys may be made by melting together Mn-Cu containing 25% of Mn, and ferro-copper containing 10% of Fe, and adding Al to the melt.

Alloys. SOC. ANON. DE COMMENTRY-FOURCHAMBAULT ET DECAZEVILLE. Brit. 159,492, Nov. 5, 1920. Addition to 140,507 (C. A. 14, 2159). In modifications of the Fe-Ni-Cr alloys described in the principal patent, the max. contents of Ni may be increased to 40%, C to 1%, and Mn to 5%. One or more other metals may be present as follows: 0.2-1% of V, 0.1-0.5% of Ti, 0.5-5% of W, 0.2-3% of Mo, and up to 10% of Co.

Heat treatment of alloys. A. DE LAVANDEYRA. Brit. 159,852, Aug. 24, 1920. The hardness and tensile strength of alloys is increased by supplying heat to the material at a substantially const. temp., detd. pyrometrically, when the critical temp is reached continuing heating until the temp. is a little above the critical point, and then quenching. In treating an alloy consisting principally of Al and Cu having a critical temp. of 510-520°, the charge is heated in a metal or saline heating bath and the temp. is indicated by a recording pyrometer. After the critical temp. is reached, the material is left in the furnace, which is arranged to be at a temp. of about 530° for about 30 min. so as to raise the temp. of the material some 10 or 15° above the critical temp. The material is then quenched, preferably in oil, for 5-15 min. and then allowed to stand in hot H_2O for about 1.5 hr.

Solders. A. H. PHILLIPS AND C. G. ARNOLD. Brit. 159,797, July 13, 1920. A solder, particularly for cast Fe, consists of about 2.2% of Co, 10% of Cu, 0.6% of Ag, 0.6% of Sb, and 86.6% of Sn.

Soldering metal. HANS CARL GROSSPETER AND WILHELM SCHUEN. Ger. 328,608, Mar. 11, 1917. The soldering metal consists of an alloy of Na with soft metals, such as Pb, Sn, Pb-Sn or the like.

Soldering aluminium. A. PASSALACQUA. Brit. 159,480, Feb. 25, 1921. In soldering Al or Al alloys by means of pure Sn and a soldering Fe, there is applied to the cleaned parts prior to soldering a liquid compn. made by mixing together a paste of linseed oil, olive oil, resin, paraffin and solid fat, and aq. soln. of nickel sulfate, NH_4Cl , and $\text{Na}_2\text{P}_2\text{O}_7$, and an aq. soln. of SnCl_2 , $\text{Na}_2\text{P}_2\text{O}_7$ and citric acid, the paste and the two aq. solns. being separately prepd.

10—ORGANIC CHEMISTRY

C. A. ROULLER

Friedr. Gustav Karl Emil Erlenmeyer, 1864–1921. B. LEPSIUS. *Ber.* **54A**, 107–13 (1921).—An obituary with a bibliography of Erlenmeyer's work. E. J. C.

Martin Freund. L. SPIEGEL. *Ber.* **54A**, 53–79 (1921).—An obituary contg. a review and bibliography of Freund's work. E. J. C.

Recent advances in stereochemistry. B. K. SINGH. *Proc. 7th Indian Sci. Congress.* Presidential Address.—A general discussion of the relation between optical activity and chem. constitution. The effects of homology and conjugated unsatn. on optical activity are taken up in detail. No new exptl. data are given. CARL S. MARVEL

A new method for the purification of methanol. A. LANZENBERG AND J. DUCLAUX. *Bull. soc. chim.* [4] **29**, 135–6 (1921).—MeOH and CHCl_3 form a const. boiling mixt. that dists. at 53° , and acetone and CHCl_3 form a mixt. that dists. at 64° . Technical MeOH may be purified by making use of these facts. A mixt. of 1 part technical MeOH and 7.5 parts of CHCl_3 when distd. gave a fraction, b. $52.5\text{--}53.5^\circ$. The MeOH was extd. by washing this fraction 3 times with an equal vol. of water and finally obtained pure by distg. the water soln. A second distn. gave nearly anhydrous MeOH that contained only 0.27% of acetone. The yield of pure MeOH is 70% of the technical MeOH used. CARL S. MARVEL

New technological applications of old reactions—Ethyl and isopropyl alcohols from hydrocarbons. "K." *Chem. Age* (London) **4**, 440–3 (1921).—Discussion of the synthesis of iso-PrOH from C_3H_8 , produced by cracking gasoline, the fundamental reactions of which were studied by Faraday 100 years ago and investigated by Hennell in 1826, and of EtOH from AcH, described by Berthelot in 1860. C. J. WEST

β, β' -Dichlorodiethyl disulfide. GEORGE MACDONALD BENNETT. London. *J. Chem. Soc.* **119**, 418–25 (1921).—The behavior of S_2Cl_2 and C_2H_4 has been the subject of some controversy, the issue being whether a monosulfide or a disulfide is formed. Bennett has reinvestigated the reaction at $30\text{--}35^\circ$ and concludes that the liquids are two-phase systems in which the continuous phase is nearly pure $\text{S}(\text{C}_2\text{H}_4\text{Cl})_2$ and the disperse phase a liquid consisting chiefly of S. The varying behavior on diln. with Et_2O , etc., may be due to different degrees of dispersion. 50 cc. S_2Cl_2 gave 111 g. of a pale amber-colored liquid, $d_{15} 1.373$, m. $5\text{--}6^\circ$; the relative viscosity at 15° was 10.25, which is characteristic of liquid-liquid 2-phase systems. The product b₁₀ $110\text{--}20^\circ$. The addition of 20 cc. dry Et_2O to 30 cc. of the liquid before distn. produced no sepn. into 2 phases. 100 cc. Et_2O caused the sepn. of 4.7 g. of a S-yellow oil, which was impure S. Heating in a stream of dry air for an hr. also caused the sepn. of S. In each case the m. p. of the sulfide was unaffected. Attempts to prep. the emulsion from the sulfide and S were unsuccessful. $\text{SHCH}_2\text{CH}_2\text{OH}$ (*Ann.* **124**, 257) was prepd. by melting 225 g. cryst. Na_2S on the H_2O bath, satg. with H_2S , cooling to 50° and stirring in 70 g. $\text{ClCH}_2\text{CH}_2\text{OH}$. It b₁₅ 58° , $d_4^{20} 1.1153$, $n_D^{20} 1.4443$. This was oxidized to β, β' -dichlorodiethyl disulfide $\text{C}_4\text{H}_8\text{Cl}_2\text{S}_2$, by H_2O_2 , FeCl_3 or NaBrO , straw-colored oil, b₁₀ 155° , $d_4^{20} 1.3375$, $(\eta/\eta_{\text{H}_2\text{O}})_{20}$ 6.08. It resembles the monosulfide in many of its properties. Its vesicant action was $1/2$ that of the monosulfide. Boiled with PbONa , it yields β, β' -diphenoxydiethyl disulfide, small, silvery plates, m. $96\text{--}7^\circ$. Na_2S gives $\text{S}(\text{C}_2\text{H}_4)_2\text{S}$. On oxidation it yields $\text{C}_2\text{H}_4\text{O}_2\text{ClSNa.H}_2\text{O}$ (*Ann.* **223**, 213). C. J. WEST

The interaction of sulfur monochloride and substituted ethylenes. WILLIAM JACKSON POPE AND JAMES L. B. SMITH. Univ. Cambridge. *J. Chem. Soc.* **119**, 396–400 (1921).— S_2Cl_2 does not react with C_2HCl_3 as with C_2H_4 , but acts merely as a chlorinating agent. The homologs of C_2H_4 , however, act in the same way as C_2H_4 , and the equation for the formation of mustard gas is a general reaction for the ethylenic hydrocarbons. β, β' -Dichlorodipropyl sulfide $(\text{CHMeClCH}_2)_2\text{S}$, results by the absorption of C_2H_4 by

S_2Cl_2 . The reaction is attended by the evolution of a large amt. of heat, and the temp. must be controlled. Below 70° , the product is a pale yellow oil, while between 80 and 100° , a dark colored oil results. Both deposit S on cooling, and the sulfide b₁₁ $105-6^\circ$. It has a marked vesicant action. On oxidation, neither the sulfoxide nor the sulfone would cryst. On prolonged oxidation, and addition of $BaCO_3$, barium β -chloropropenesulfonate is formed, scales, $(CHMeClCH_2SO_3)_2Ba \cdot 2H_2O$. β, β' -Dichlorodisecbutyl sulfide, b₁₁ $121-22^\circ$, is almost without vesicant action. The sulfone, viscous oil, is converted to a resinous mass on immersion in boiling liquid air. Barium β -chlorobutane- γ -sulfonate, $(ClCHMeCHMeSO_3)_2Ba \cdot 2H_2O$, scales. On boiling the sulfide with alc. Na_2S , the sym. tetramethyldiethylene disulfide, $S(CHMeCHMe)_2S$, is obtained, oil with odor of decaying onions, b₁₈ $145-50^\circ$. The rate of absorption of the three hydrocarbons is shown in the following table:

Time.	C_3H_4	Volume absorbed, C_3H_4	C_4H_6
0.4	—	5.5	26.
0.8	—	23.0	40.2
1.0	0.3	33.0	42.3
1.5	0.6	43.0	43.2
2.0	1.0	43.8	Ended
3.0	2.7	Ended	
4.0	6.7		
5.0	23.0		
6.0	43.8		

The catalytic effect observed in the case of C_3H_4 is absent in the absorption of the other hydrocarbons. C. J. WEST

Sulfur-containing derivatives from ethylene chlorohydrin. EMIL FROMM AND ADOLF KORN. Univ. Freiburg i. B. Ber. 54B, 320-6(1921).— ω, ω' -Dibenzoyloxydiethyl sulfide, from $(HOCH_2CH_2)_2S$ shaken with $BzCl$ and $NaOH$, care being taken that the temp. does not rise and that the liquid remains permanently alk., crystals from dil. alc., m. 65° ; 10 g. in $AcOH$ treated with 3.5 g. of 30% H_2O_2 and poured into H_2O after 12 hrs. gives about 58% of the sulfoxide, needles from bot $EtOH-H_2O$, m. $83-5^\circ$. Sulfone, obtained in 78% yield from the sulfide shaken in $AcOH$ with satd. $KMnO_4$ and dil. H_2SO_4 until the red color is permanent, silvery leaflets from dil. alc., m. 95° . Sulfide dibromide, $SBr_2(CH_2CH_2OBz)_2$, from 2 g. of the sulfide and 1 g. Br in ice-cold ligroin, yellow needles from C_4H_6 , m. 81° , slowly decomp. in moist air, gives the sulfoxide when shaken with H_2O . Diiodide, dark red crystals obtained by rubbing the components dry or hinging them together in ice-cold ligroin. ω -Hydroxyethyl p -tolyl sulfide (A), obtained in 83% yield from 40 g. $p-MeC_6H_4SH$ in concd. $NaOH$ cautiously warmed on the H_2O bath with 25 g. $HOCH_2CH_2Cl$ and heated 2 hrs. longer after the vigorous reaction has ceased, b. $282-3^\circ$ (decompn.), b₁₀ 174° ; allowed to stand in $AcOH$ with a slight excess of 30% H_2O_2 24 hrs., freed from the excess of H_2O_2 with H_2S , filtered, evapd. at $40-50^\circ$, taken up in Et_2O and washed with H_2O , it yields the sulfoxide, which cannot be distd. but by the Schotten-Baumann method gives the benzoate, m. 106° , described below. When it is attempted to dist. the crude sulfoxide it decomp. 100° and there dist. at about 192° under 12 mm. substances which partially solidify and contain the sulfone, as shown by the formation of small amts. of the benzoate, m. 170° (see below). Sulfone, obtained in 80% yield from 25 g. $p-MeC_6H_4SO_2Na$ in a little H_2O and a few drops dil. $NaOH$ at about 60° treated with 15 g. $HOCH_2CH_2Cl$ and heated 4 hrs. on the H_2O bath under a reflux, needles, m. 39° , b₁₁ 230° . Benzoate of A, needles, m. 21° , b₁₁ 233° . Benzoate of the sulfoxide, from 5 g. of the benzoate of A allowed to stand 12 hrs. in $AcOH$ with 2.5 g. of 30% H_2O_2 , needles from dil. alc., m. 106° , gives with an equal

wt. of Br in cold AcOH an unstable yellow *dibromide*, which quant. regenerates the sulf-oxide with H_2O . *Benzoate* of the sulfone, from the benzoate of A with cold $KMnO_4$ and H_2SO_4 , rod-shaped crystals from alc., m. 171° , more easily prepd. by benzoylating the sulfone. *ω -Benzoyloxyethyl *p*-tolyl sulfide dibromide*, from the components in AcOH or ligroin, yellow substance, m. 101° , hut decomps. in the presence of H_2O , becoming white and evolving HBr. *Diiodide*, from the sulfide and the calcd. amt. of I heated 4 hrs. in $CHCl_3$ on the H_2O bath under a reflux, wine-red crystals from C_6H_6 , m. $58-70^\circ$, decomp. with sepn. of I on heating or long standing, also obtained by rubbing the dry components together, converted into the sulfoxide by heating with $AgOAc$ in AcOH. *ω -Chloroethyl *p*-tolyl sulfide*, from a boiling solu. of A treated 4 hrs. with HCl (yield, 75%), yellowish liquid, b_{72} 150° , h. $255-7^\circ$, also obtained by boiling A with HCl under a reflux and, less satisfactorily, by the action of PCl_5 , is very toxic. *Sulfoxide*, dark liquid, which decomps. on distn. *in vacuo*. *Sulfone*, crystals from 50% alc., m. 71° . *ω -Iodoethyl *p*-tolyl sulfide*, from the Cl compd. heated with KI in alc. under a reflux, reddish oil.

C. A. ROUILLER

Preparation of amines of secondary alcohols. ALPHONSE MAILLIE. *Bull. soc. chim.* 29, 219-23 (1921); *Compt. rend.* 172, 692-4 (1921).—M. has shown (C. A. 14, 2336) that the hydrogenation of ketazines gives a mixt. of primary and sec. amines. The reaction is now further applied to the azines of unsymm. ketones. $MeCOCHMe$, b. 93.5° , boiled 6 hrs. with $N_2H_4 \cdot H_2SO_4$ and NaOH in alc., gives the *ketazine*, $(Me_2CHMeCH:N)_2$ (A), b. 165° . A, hydrogenated as vapor over Ni at $180-200^\circ$, gives NH_3 and a liquid containing primary and (more) sec. amine; $Me_2CHCHMeNH_2$, b. $76-8^\circ$, absorbs CO_2 and gives the carbylamine reaction; with $PhNCO$, forms a *phenylurea*, needles, m. 144° ; $(Me_2CHCHMe)_2NH$, b. $178-80^\circ$, and forms a cryst. HCl salt. Similarly, $PrCOMe$ gave an *azine*, b. $198-200^\circ$; *primary amine*, b. $86-8^\circ$ (*phenylurea*, m. 115°); *secondary amine*, b. $185-8^\circ$. Me_2CHCH_2COMe yielded the *ketazine*, b. 178° ; *primary amine*, b. $108-10^\circ$ (*phenylurea*, m. 108°); *secondary amine*, b. $208-10^\circ$. $Me_2CHCH_2CH_2COMe$ gave a *ketazine*, b. $155-60^\circ$; *primary amine*, h. 137° (*phenylurea*, m. 103°); *secondary amine*, b. 240° . $Me_2CHCH_2COCHMe_2$ yielded a *ketazine*, b. $190-5^\circ$; *primary amine*, b. $145-8^\circ$; *secondary amine*, b. 245° . The yield of primary amine is greater as the complexity of the original ketone increases.

BEN H. NICOLET

The chlorovinylchloroarsines. STANLEY JOSEPH GREEN AND THOMAS SLATER PRICE. *J. Chem. Soc.* 119, 448-53 (1921).— C_2H_2 was passed into a cooled mixt. of 440 g. $AsCl_3$ and 300 g. $AlCl_3$; after 6 hrs. 100 g. C_2H_2 had been absorbed. The product was decompd. with cold HCl and the resulting oil fractionated, giving the 3 chloroarsines: *chlorovinylchloroarsine*, $C_2H_2Cl_2As$, b_{26} 93° , b_{30} 96° . It is a very marked vesicant and a very powerful respiratory irritant. On adding Br in CCl_4 , a *bromo compound*, m. 122° , seps. The 2nd fraction is *dichlorodivinylchloroarsine*, $C_4H_4Cl_2As$, b_{24} $130-3^\circ$. It has much less marked vesicant properties than the dichloride but it is a more intense respiratory irritant. On oxidation with HNO_3 , a cryst. product is obtained, m. 97° . The 3rd product was *trichlorodivinylarsine*, $C_4H_4Cl_3As$, b_{24} $151-5^\circ$, m. $3-1^\circ$. While neither a strong vesicant nor a powerful respiratory irritant it induces violent sneezing. It is characterized by being insol. in alc. This substance is the main product of the reaction, but it can be easily and quant. changed to a mixt. of the primary and secondary chloroarsines by heating at $200-250^\circ$ with $AsCl_3$. In the absorption expt., a solvent, CCl_4 , may be used, the yield of the chlorides being about 65% in either case. The secondary and tertiary arsines tend to form very easily and at the expense of the primary. Varying the proportions of $AsCl_3$ and $AlCl_3$ between wide limits produced little difference in the course of the reaction. $FeCl_3$ behaved similarly hut less energetically.

C. J. WEST

Experiments on the production of compounds containing arsenic as a center of optical activity. GEORGE JOSEPH BURROWS AND EUSTACE B. TURNER. Univ. Sidney J.

Chem. Soc. 119, 426-37 (1921).—Definite evidence is presented that As compds. may be resolved into optically active components, but the active compds. racemize so rapidly that the observed rotation was small. This racemization appears to occur as the result of the tendency of the quaternary As salt to dissociate readily in soln. Me_3AsI is readily prepd. by dissolving 320 g. As_2O_3 , 206 cc. MeI and 388 g. NaOH in aq. alc. and after 20 hrs., distg. off the alc., acidifying with HCl and satg. with SO_2 . γ -Phenylpropyldimethylarsine (A), $\text{C}_{11}\text{H}_{17}\text{As}$, by adding 35 g. Me_3AsI in 100 cc. C_6H_6 to the Grignard reagent from 40 g. $\text{PhC}_6\text{H}_4\text{Br}$, 5 g. Mg and 100 cc. Et_2O , highly refractive liquid, b_{14} 133°. *Methiodide*, needles, m. 144°. *Additive compound*, $\text{C}_{11}\text{H}_{17}\text{As} \cdot \text{Me}_3\text{AsI}$, prisms, m. 78-81°, which completely dissociates in C_6H_6 . No other arsine examd. forms this addition product. γ -Phenylpropylmethylchloroarsine (B), $\text{C}_{10}\text{H}_{14}\text{AsCl}$, by the action of Cl upon A in CCl_4 , b_{14} 164-7°. *Bromo compound*, b_{14} 177-80°. 5 g. B and 2.7 g. AlCl_3 , boiled in CS_2 for 3 hrs., gave 2 g. *asym-methyltetrahydroarsinoline*, $\text{C}_{10}\text{H}_{12}\text{As}$, b_{14} 140°, with an odor suggestive of quinoline. *Methiodide*, prisms, m. 235°. A hot alc. soln. has a yellow color, which disappears on cooling. *Chloroplatinate*, yellow, microcryst. powder, m. 170°. *Picrate*, yellow. *Picrolonate*, yellow. Phenyl- γ -phenylpropylmethylarsine, $\text{C}_{16}\text{H}_{19}\text{As}$, by the action of $\text{PhC}_6\text{H}_4\text{MgBr}$ upon PhMeAsI , highly refractive liquid, b_{17} 208°. *Methiodide*, rhombohedra, m. 102°. Phenyl- α -naphthylmethylarsine, $\text{C}_{17}\text{H}_{15}\text{As}$, from 117 g. PhMeAsI , 94 g. $\text{C}_{10}\text{H}_7\text{Br}$ and 14 g. Mg , m. 58°, b_{17} 236-8°. *Methiodide*, plates, m. 175°. The oxide forms well defined prisms, m. 175°. Hydroxyphenyl- α -naphthylmethylarsonium *d*- α -bromocamphor- α -sulfonate, formed in AcOEt soln., m. 161°. Attempts to fractionate this into 2 diastereoisomerides gave very small differences in mol. rotation and the results were regarded as insufficient evidence upon which to base the assumption that As was functioning as a center of optical activity in this compd. Methylthylidoursine, $\text{C}_3\text{H}_4\text{IAS}$, from 250 g. EtAsI_2 , 122 g. NaOH in 200 cc. H_2O and 600 cc. alc., and 75 cc. MeI , yellow, oily liquid, b_{14} 65°. Phenyl- α -naphthylmethylarsonium bromide, $\text{C}_{20}\text{H}_{19}\text{BrAs}$, m. 140° (decompu.). Phenacylphenyl- α -naphthylmethylarsonium bromide, by warming $\text{Ph}(\text{C}_6\text{H}_5)\text{MeAs}$ and BzCH_2Br , m. 189°. Homopiperonylphenyl- α -naphthylmethylarsonium bromide, $\text{C}_{24}\text{H}_{23}\text{O}_2\text{BrAs}$, plates, m. 174-5°. *d*- α -Bromocamphor- α -sulfonate, $\text{C}_{10}\text{H}_{15}\text{O}_4\text{BrAsSC}_6\text{H}_5\text{O}$, stout needles. This could not be obtained pure and so fractionation expts. were of little value. Phenyl- γ -naphthylbenzylmethylarsonium bromide, prisms, m. 185°. *d*- γ -Bromocamphor- α -sulfonate, colorless prisms from EtOH - AcOEt , acetone or acetone- Et_2O , m. 187°, $[\text{M}]_D^{25}$ 281°. On recrystn. this was raised to 300°. The *arsonium iodide*, $\text{C}_{14}\text{H}_{15}\text{IAS}$, forms needles, m. 186-7°, $[\text{M}]_D^{25}$ 12°. Recrystd., the product was inactive. The *d*-bromide m. 187-8°, but racemization in soln. was rapid and the highest value found for $[\text{M}]_D^{25}$ was 5°. From studies on the formation of quaternary arsonium compds. it is concluded improbable that those containing 2 similar radicals can exist in more than 1 form.

C. J. WEST

The stability of tautomeric formaldehydephenylhydrazones. NEVIL VINCENT SIDGWICK AND ELINOR KATHERINE EWBANK. Oxford. *J. Chem. Soc.* 119, 486-492 (1921).—Bamberger and Schmidt (*Ber.* 34, 574, 2001) infer that the stability of a series of substitution products of $\text{CH}_2\text{:NNHPh}$, which occur in 2 forms, both in the solid state and in soln., is detd. by the solvent with which it is in contact. This has been shown to be incorrect in 2 cases, BzCH:NNHPh and $\text{O}_2\text{NCH:NNHPh}$. In the 1st case the α -form, m. 111-2°, was obtained from C_6H_6 or petr. ether. When this form is digested with alc. several hrs. at 40-55° or several days at room temp., the β -form results, m. 132-3°. When distd. with steam and then digested with alc., the β -form m. 145-6°. After standing 1 month in either alc. or petroleum ether, the α -form was converted into the β -form. The peculiar behavior of the substance must be due to the fact that the change to the β -form is slow. The soly. of the α - and β -forms at 32° is: C_6H_6 , 25.1%, 8.0; heptane, 1.7, 0.55; CCl_4 , 8.8, 2.27; cyclohexane, (2.58), 1.66. In

C_6H_6 at 3° , 5.84 and 0.32%. Soly. data are given for other temps. also, in EtOH, C_6H_6 , and heptane. From the figures it follows that in C_6H_6 soln. at 3° , there is at equil. approx. 4 times as much α - as β -form. The value of G (the van't Hoff-Dimroth constant) at 3° is calcd. to be 4.5. The instability of $NO_2CH:NNHPh$ made an extensive investigation impossible, but the results show that the β -form of the hydrazone is stable in contact with non-associated as well as with associated solvents. It may be assumed that the same is the case with the other hydrazones examd. by B. and S. C. J. WEST

The manufacture of methyl ethyl ketone by catalysis. JEAN TOUSSAINT. *J. pharm. Belg.* 3, 221-224 (1921).—MeEtCO is prepd. on a com. scale as follows: Normal BuOH, obtained as a by-product in the fermentation of grain, is converted into EtCH:CH₃ by passing the vapors over Al_2O_3 heated to 360° . The gaseous C_4H_8 is liquefied by subjecting it to a temp. of -5° , placed in a metallic cylinder and agitated with H_2SO_4 . The EtCH(HSO₄)Me, thus formed, is converted into EtCH(OH)Me by dilg. with H_2O and distg. To obtain MeEtCO, the BuOH is distd. and the vapors are passed over finely divided Cu heated to 250° . A. G. DUMÉZ

Determination of the constitution of glycerides. A. D. GRÜN AND FRANZ WITTEK. Georg Schicht A. G., Aussig a. d. E. *Ber.* 54B, 273-89 (1921).—In view of the fact that in many cases there is possibly no synthetic method by which the structure of glycerides can be established beyond doubt, G. and W. have attempted to solve the problem by another method, viz. by oxidation or substitution reactions of the mono- and diglycerides, which could be extended to the triglycerides by a combination of partial degradation to di- and monoglycerides and detn. of the structure of the degradation products by the new method. Since this work was begun, the synthesis of glycerides of definite structure has been greatly extended by Fischer and his coworkers (cf. C. A. 15, 684), but G. and W. do not believe that their own results have become entirely superfluous. The quant. detn. of C:O compds. in the oxidation products was effected by the Benedikt-Strache method as improved by Kaufer-Smith and by Meyer (*Analyse organischer Verbindungen*, Berlin 1916, p. 701), which consists in treating with a measured excess of PhNHNH₂, oxidizing the excess with Fehling soln. and measuring the N evolved. Blank expts. showed that under the conditions the PhNHNH₂ does not react with the glycerides themselves. Five g. synthetic distearin, m. after a long time at 74.5° , prepd. by Kraft and Guth's method from $HOCH(CH_2Cl)_2$ and K stearate, was treated in 150 cc. AcOH at 35° with 1.8 g. powdered $KMnO_4$ in the course of 6 hrs., poured into H_2O , shaken with $(CO_2H)_2$ in 2 N H_2SO_4 until the Mn oxides had dissolved, extd. with $CHCl_3$, washed, dried, freed from the $CHCl_3$ at 30° by means of CO_2 and shaken 1 hr. in Et₂O with charcoal; the neutral soln. yielded 4.5 g. of a cryst. product identified by its m. p. (74.5°), mixed m. p., sapon. no. (182.0) and negative C:O test as the original distearin, and 0.3 g. of a second fraction with 0.93% C:O (calcd. for $CO(CH_2)_2CCl_2H_{11}$) (A), 4.51%, corresponding to about 20% of a C:O compd. which must have been a ketone as the product gave negative qual. tests for aldehydes (NH_3 , $AgNO_3$, fuchsin- SO_2 , diazobenzenesulfonic acid, etc.). In a second expt. with twice as much $KMnO_4$ (100% excess) the first fraction, of only 2 g., m. 73° , sapon. no. 181.7, C:O content 0.01%, and the second fraction, 2.5 g., m. 68° , sapon. no. 183.4, C:O content 1.25%, indicating that it consisted of a mixt. of distearin and A in the ratio of about 3:1, with a very small amt. of distearylglyceric ester. A comparison of these results with those obtained in the oxidation of other distearins, where considerable amts. of the ester and also free acids were always obtained, shows that the compd. under investigation was α, α' -distearin mixed with a very little of the α, β -isomer. A synthetic distearin prepd. from α, β -distearo- α' -chlorohydrin by means of $AgON_3$, which m. 77.5° , gave, when oxidized as above, besides a little unchanged distearin and 0.4 g. acid products, 1 g. m. 79° , sapon. no. 183.6, C:O content 2%, shows no aldehyde reactions, and 1.7 g. m. at the temp. of the haud, sapon. no. 224.3, contains no C:O. The first

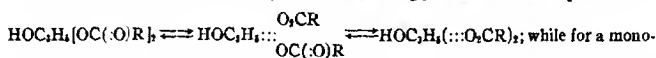
fraction was, therefore, a mixt. of about equal parts of unchanged distearin and A and the second fraction is believed, in spite of its low m. p., to have been distearyl distearyl-glycerate, $\text{RCH}_2\text{CHRCO}_2\text{CH}_2\text{CHRCO}_2\text{R}$ (B) ($\text{R} = \text{C}_{17}\text{H}_{35}\text{CO}_2$). Assuming that the distearin component of the ester and the free acids (as well as the esterified acid) produced in the oxidation are derived from the α,β -compd. while the distearin forming the mixed crystals with the C:O compd. is the α,α' -compd., the original distearin must have consisted of approx. equal parts of α,α' - and α,β -distearin. It has been shown (C. A. 6, 1687) that by the stepwise sapon. of triglycerides with concd. H_2SO_4 it is possible to prep. diglycerides, which, from their m. ps. and mixed m. ps. and the ease with which the HO is replaced by Cl, were concluded to be the unsym. compds. To test these conclusions 200 g. powdered com. tristearin thoroughly mixed with 70 g. of 100% H_2SO_4 was quickly fused, heated to 70° , at once poured into 2 l. of boiling 2% Na_2SO_4 , treated with 50 g. NaCl, removed from the flame and allowed to stand 5-10 min. and the clear fat layer washed free from mineral acids with salt water, dried with Na_2SO_4 dissolved in an equal amt. of alc., neutralized cold with weak alc. alkali, freed from alc., treated with CaCl_2 dust and a little soda, dried and extd. with Me_2CO and the ext. fractionally crystd. from alc., Et_2O and Me_2CO and mixts. of these solvents. A total of 3500 g. tristearin yielded about 1270 g. of glyceride mixt., from which were obtained only about 11 g. mono- and 340 g. distearin in approx. pure form. Assuming that the mixed fractions consisted each of only 2 glycerides (mono- + di- and di- + tristearin), their compn. can be calcd. from the sapon. and HO nos.; the yields, including the pure fractions, were then 29.7 g. mono-, 1011 g. di- and 228 g. tristearin. As the mean sapon. no. of the crude product was almost the same as that of distearin, the crude product must have contained much more monostearin than indicated above, viz. 2 parts to 5 parts tristearin, and a large part of it must have been decompd. in working up the crude product, probably in the neutralization. In one case there was isolated from the low-melting glyceride mixt. an apparently pure compd., leaflets, m. 56° , sapon. no. 179.6, m. 66° after standing 1 year, which is possibly a lower melting modification of a distearin (cf. Berthelot, *Ann. chim. phys.* [3] 41, 420 (1854)). Oxidation of 10 g. of the distearin as above yielded 0.5 g. acids, 2.5 g. of the mixt. of distearin and A and 3 g. distearyl distearyl-glycerate; of the last, about 0.5 was obtained in fine scales m. 68° , sapon. no. 233.3, the other 0.5 as the soft, low-melting cryst. mass described above, sapon. no. 233.0. In a second expt. with a 50% excess of KMnO_4 (2.7 g. for 5 g. distearin) were obtained 0.3 g. acids and 4 fractions of neutral substances: 1 g. of pure distearin, 2 g. of a mixt. of about equal parts distearin and A (C:O content 2.4%), and 3.5 and 2.5 g. fractions consisting of distearin with 20% and 2%, resp., of C:O compds. and a very little ester. Oxidation of the low-melting distearin gave 20% acids and 80% neutral substances. Of the former about 0.5 corresponds, from the acid no. (147.3-149.2), to monostearylglyceric acid and the other 0.5, from the sapon. no. (269.8), to distearyl-glyceric acid. The neutral products all consisted of mixts. in which A predominated; the chief fractions contained 3.40 and 2.82% C:O, resp.; B was present only in small amt.; tristearin could not be detected with certainty. The results do show with certainty, however, that among the products of cleavage of tristearin with H_2SO_4 there are considerable amts. of α,α' -distearin; whether it is a primary degradation product or, which is more probable, is a secondary product, formed by synthesis from glycerylsulfuric acid and stearic acid or by rearrangement of the α,β -isomer, cannot be detd. The monostearin obtained by the degradation of tristearin with H_2SO_4 sep'd. from $\text{EtOH-Et}_2\text{O}$ in small cryst. aggregates, from Et_2O -petr. ether as a cryst. meal, m. 80° ; 5 g. in 50 cc. AcOH at 30° with 6 g. KMnO_4 gave 1 g. unchanged monostearin and 4 g. acid, from which by further crystn. was obtained 3 g. pure *stearylglycolic acid* (C), needles, m. 89° . As α -monostearin should yield first $\text{C}_{17}\text{H}_{35}\text{CO}_2\text{CH}_2\text{COCO}_2\text{H}$ and β -monostearin should give $\text{C}_{17}\text{H}_{35}\text{CO}_2\text{CH}(\text{CO}_2\text{H})_2$ (D), both of which would then

yield C, the oxidation method is not applicable to the detn. of the structure of mono-glycerides. From 4 g. of a monostearin prepd. by the stepwise alcoholysis of tristearin (200 g. from 3000 g. tristearin) in 40 cc. AcOH with 4.8 g. KMnO_4 , were obtained 0.8 g. unchanged monostearin and 2.9 g. pure C. C can be obtained in 9.3 g. yield from 5 g. $\text{ClCH}_2\text{CO}_2\text{H}$ and 15 g. stearic acid, each in 20 cc. alc., neutralized with alc. KOH, mixed, evapd., heated 6 hrs. at 110° , rubbed with a little alc., then with H_2O , acidified with H_2SO_4 , taken up in Et_2O and repeatedly crystd. from Et_2O and Me_2CO ; the mother liquors yield 4.5 g. of a stable addition product of C with stearic acid, $\text{C}_{32}\text{H}_{74}\text{O}_4$, leaflets, m. 76° . Attempts were made to synthesize D by shaking 1.2 g. tartaric acid in 30 cc. $\text{C}_6\text{H}_5\text{N}$ with 3.5 g. stearyl chloride for 5 hrs., taking up the upper layer in Et_2O , washing free of $\text{C}_6\text{H}_5\text{N}$, drying and concg. at a low temp. by means of a current of CO_2 or over P_2O_5 , H_2SO_4 or oil, but in concd. solu. CO_2 is always split off and the products which sep. are mixts. of D with C and stearic acid, the last two of which can be crystd. out but the D could never be isolated. One g. of the monostearin from $\text{ClCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH}$ and K stearate heated 10 min. at 115° with 0.5 g. PhNCO and allowed to stand 2 days gives almost quant. (1.3 g.) the diphenylurethan, while the monostearin obtained by stepwise degradation of tristearin with H_2SO_4 or by stepwise alcoholysis gives, together with only about 0.2 g. of the diphenylurethan, chiefly $\text{CO}(\text{NHPh})$.

CHAS. A. ROULLER

Interchange of ester radicals ("Umesterung") and its relation to the constitution of the fats. AN. GRÜN, WITH FRANZ WITTKA AND JOSEF SCHOLZE. Georg Schicht A.-G., Aussig a. d. E. Ber. 54B, 290-9 (1921); cf. Fischer, C. A. 15, 842.—Some of the observations reported by Fischer have long been known and the significance of the interchangeability of ester radicals as a general reaction, at least in the field of the fats, has not been so ignored as he assumes. There are not only numerous communications on ways of carrying out the reaction other than those described by him and on its analytical, preparative and even technical applications, but there have even been attempts to give a theoretical explanation of it (*Öl- u. Fettind.* 1, 225 (1919)). The possibility suggested by F. of the interaction of glycerol with the esters of monohydric alcs. in the absence of catalysts has been realized, as described in the present paper, as well as the reverse reaction, the reaction of triglycerides with various monohydric alcs. Of the greatest importance in F.'s paper is his suggestion that this interchangeability of ester radicals in the esters of polyvalent alcs. may occur without catalysts at the m. p. or, more slowly, even without heating at all, and that the change in m. p. shown by many glycerides on simple fusion and resolidification and in some cases merely on long standing may be due to such an interchange of ester radicals. But while such an explanation is possible for diglycerides and mixed (polyacid) triglycerides, it does not explain similar m. p. anomalies shown by simple (monoacid) triglycerides. All these phenomena can be explained on the simple assumption that glycerides, like other carboxylic esters, may exist in coordination-isomeric forms (Hantzsch, C. A. 12, 1641), as already pointed out by G. in 1919 (*loc. cit.*); as the journal in which this paper was published is not readily accessible, G. again gives his views in somewhat amplified form. H. has shown that free CO_2H acids are equil. mixts. of pseudo acids (A), optically identical with the esters (B), and of the coordination isomers, the true acids (C), optically identical with the salts (D): $\text{RC}(\text{O})\text{OR}'(\text{B}) \longrightarrow \text{RC}(\text{O})\text{OH}$ (A) $\rightleftharpoons \text{RC} \begin{smallmatrix} \text{O} \\ \diagup \\ \text{O} \end{smallmatrix} \text{H}$ (C) $\longleftarrow \text{RC} \begin{smallmatrix} \text{O} \\ \diagdown \\ \text{O} \end{smallmatrix} \text{M}$. In the coordination form both O atoms are present in the same condition; there is an affinity compensation between them so that the H appears to be uniformly bound to both O atoms or the whole mol.; as in the inorg. acids it is in the second sphere, is bound ionogenically. As shown by H., the salts, by solvate formation, may be partially converted into the pseudo acid or ester form. Assuming that, conversely, the esters, by solvate formation or otherwise, may, at least to a small extent, be converted into the coordina-

tion form, all the above phenomena observed in the cleavage of fats, the interchange of ester radicals, the anomalies in m. ps., can be explained. In the coordination form the union between the alkyl and acyl is loosened; it is ionogenic and the alkyl is prep. for interchange with a metal or another alkyl. The existence of monoacid glycerides, like tristearin, in 2 (or more) forms can be explained by the assumption of a rearrangement into a coordination form. For each monoglyceride, 2 isomers are possible: $(\text{HO})_2\text{C}_1\text{-H}_2\text{OC}(\text{:O})\text{R} \rightleftharpoons (\text{HO})_2\text{C}_1\text{H}_2\text{:O}_2\text{CR}$; for a monoacid diglyceride there are 3 possibilities:



acid triglyceride there are 4 possible isomers: the pure ester form, the pure coordination form and the 2 mixed forms. As is well known glycerides can be quant. hydrolyzed without the aid of catalysts, such as acids or bases, if the temp. is sufficiently high and the stirring is thorough, and alcoholysis can be similarly effected, although to a lesser degree. Thus, when 20-g. portions of tristearin were heated with 20 g. abs. alc. in autoclaves at 200° for 5, 10 and 15 hrs. (unfortunately the mixt. could neither be stirred nor shaken), freed from most of the alc. by distn. and from the rest of the alc. and from the glycerol by fusing over H_2O , dried and analyzed for Et stearate by distg. under 3 mm. at 175–80°, there were found 12.7, 13.0 and 12.0%, resp., of the ester. At 270°, the yields of Et stearate were 30.6, 29.3 and 26.1%, resp., after 5, 10 and 15 hrs. With equal wts. of iso-AmOH and tristearin at 270°, 14.3 and 14.0% ester were obtained in 5 and 15 hrs., resp. As iso-AmOH is a much better solvent for stearin than EtOH while both iso-Am and Et stearates are good solvents for tristearin the following mixts. were heated 15 hrs. at 200°: 17.6 g. tristearin + 2.4 g. Et stearate + 20 g. EtOH, and 17.2 g. stearin + 2.8 g. iso-Am stearate + 20 g. iso-AmOH; the increase in Et stearate in the first mixt. was 0.4%, that of iso-Am ester in the second 17.7%; in an analogous expt. at 270° (except that in this case 30% Et stearate was added to the EtOH-tristearin mixt.) the increase of Et and iso-Am esters was 20.0 and 36.0%, resp. With 10 g. Et stearate, 10 g. tristearin and 20 g. alc. heated 15 hrs. at 270° the increase in Et ester was only 1%. As in the presence of catalysts, the alcoholysis is stepwise; the glyceride residue remaining after distg. the Et stearate formed at 270° showed an HO no. of 28.9 (calcd. for tristearin 0, for distearin 90) and was sepd. by fractional crystn. into 4 parts; the chief of these (about 40%) was almost pure tristearin, two fractions (about 40%) consisted of about equal parts of di- and tristearin, and the rest was apparently di- with a little monostearin. When Et stearate is heated with an excess of glycerol in an autoclave only a few % of glyceride are formed, even after several hrs. at 270°; as there is no mutual soly. thorough mixing is essential, but no suitable autoclave being available the following expts. were carried out in open vessels; a large part of the alc. set free, therefore, escaped so that the results cannot be compared directly with those of the expts. described above. Et and iso-Am stearates were heated with 0.5–3.0 mols. anhydrous glycerol, with extremely vigorous turbinizing, at 270–80°, the unchanged glycerol was washed out, the residue dried with Na_2SO_4 and the ester and glyceride were sepd. by distn. *in vacuo*. With glycerol and Et stearate in the ratio of about 1: 2, 67.3 and 58.5% unchanged ester and 32.7 and 41.5% glyceride were obtained after 5 and 10 hrs., resp.; the sapon. nos. of the glycerides were 179.7 and 186.5, resp. With equimol. amts. of ester and glycerol, 74.0, 47.6 and 3.9% unchanged ester were recovered after 5, 10 and 15 hrs., resp.; the sapon. nos. of the glycerides were in each case about the same (168–70). With 3 mols. glycerol, the amts. of unchanged ester are 65.2, 33.7 and 5.7%, and the sapon. no. of the glycerides 174.4, 169.8 and 156.4. For the iso-Am ester with 3 mols. glycerol the amts. of unchanged ester are 67.6, 35.2 and 0% and the sapon. nos. of the glycerides 169.4, 167.9 and 149.2. It is probable that the ester and the glycerol first form monostearin, which dissolves in the ester and reacts further with it to form di-

stearin; this is also sol. in the ester and, if there is not present a sufficient excess of glycerol, forms with it tristearin; if, however, there is a sufficient excess of glycerol, the reaction reverses itself and monostearin is formed from the distearin and glycerol.

CHAS. A. ROULLER

α -Sulfodipropionic acid. J. M. LOVÉN AND R. AHLBERG. *Ber.* **54B**, 227-9(1921).—L. had prepd. an α -sulfodipropionic acid (A), $\text{SO}_2(\text{CHMeCO}_2\text{H})_2$, by oxidation of α -thiodilactylic acid (B), $\text{S}(\text{CHMeCO}_2\text{H})_2$ (*Ber.* **17**, 2818(1884)). Later he prepd. the *d*-, *l*-, *dl*- and meso-forms of B (*Ber.* **29**, 1133(1896)) and it became of interest to det. whether on oxidation with KMnO_4 they would yield isomeric forms of A. It has been found that in every case the same A is obtained and all attempts to resolve this into optically active acids by means of an active base have failed. Either rearrangement takes place at the moment of O addition or the oxidized acids are unstable and rearrange into an equil. mixt., the cryst. acid obtained being the least sol. or the equil. being established in its favor. The assumption of a rearrangement until a certain equil. is established gains in probability from the fact that in the ester the H atoms on the asym. C atom can react with Na alcoholate and MeI and are, therefore, not so firmly bound. To carry out the oxidations, the B was neutralized with soda and treated with the calcd. amt. of 3% KMnO_4 in a current of CO_2 . Not only were all the products optically inactive and showed the same m. p., 170-2°, even when mixed, and the same physical appearance, but they all had the same cond. The neutral α -phenethylamine salt seps. in thick prisms, possibly with 1 H_2O .

CHAS. A. ROULLER

α -Bromocaprolylurea and its lineal homologs. M. TIFFENEAU AND E. ARDELY. *Bull. sci. pharmacol.* **28**, 155-60(1921).—The opinion is expressed that the branching of the C chains in types of compds. producing hypnosis is a factor which favors their selective action on nerve tissue, quite probably by increasing their soly. in the lipoids. On the other hand when $\text{C}_6\text{H}_{13}\text{BrCONHCONH}_2$ is administered to dogs by esophageal sound in doses of 0.4 g. per kg. no hypnosis occurs, although the Br content of the blood increases markedly. About 1/4 of the ingested Br can be recovered in the urine up to the 8th day. Similar studies with $\text{C}_8\text{H}_{17}\text{BrCONHCONH}_2$ and $\text{C}_{10}\text{H}_{21}\text{BrCONHCONH}_2$ showed the same marked retention and delayed excretion of the Br, while no hypnosis was produced, thus making evident the fact that the linear homologs of high mol. wt. do not possess the hypnotic powers of the lower homologs with branched chains.

F. S. HAMMETT

Simple cyanogen and cyanuryl compounds. II. Cyanuryl triazide (C_3N_9). ERWIN OTT AND ERNST OHSE. *Univ. Münster i. W. Ber.* **54B**, 179-86(1921); cf. *C. A.* **13**, 2533.—From 6.2 g. cyanuryl chloride added in very small portions in the course of 1-2 hrs. to 10 g. NaN_3 in 60-70 cc. ice H_2O , allowed to stand 2-3 hrs., washed several times with H_2O and dried on paper in the air is obtained 85-9% of cyanuryl triazide (A), m. 94° after 1-2 crystns. from abs. alc. By using 5 g. NaN_3 in 20 cc. H_2O and 30 cc. Me_2CO the chloride (3.1 g.) can be added in one portion and after heating 5-10 min. at 40-50° with vigorous stirring and allowing to evaporate in a flat dish a 95% yield can be obtained. The crystals are very lustrous, highly refractive and very voluminous; on melting they shrink in vol. to about 1/3 and after resolidifying have $d_{13}^{25} 1.54$. If the capillary containing the melted A is held in the flame it explodes with a violet light and a loud report. While A undergoes no change if kept a long time at 100° it explodes on long heating at 150-60° or after a short time at 170-80° or when struck, but the sensitiveness is not greater than that of Hg fulminate. To cause the explosion of a thin layer between 2 steel blocks a 100-g. wt. had to be dropped on the upper block from a distance of 15 cm. It is completely hydrolyzed to HN₃ by heating 5 min. at 50° with 0.1 N NaOH. When a sample is fused on a glass plate around which is wound an Fe wire and is then exploded in a bottle evacuated to 1 mm. by passing an elec. current through the wire, it is quantitatively converted into (CN)₂ and N₂. Attempts were made to prep. A by diazotizing the tri-

hydrazide (Finger, C. A. 1, 1548) but it was found that the diazotization of the 2nd and especially of the 3rd hydrazide grouping is so difficult that pure A cannot be obtained in this way; the substance richest in N which could be obtained contained only 82.0-2.1%; calcd. for A, 82.36%. The relatively slight reactivity of A suggests that instead of being a triazide it possibly has the structure of the isomeric cyanuryltritetrazole, but O. and O. are still inclined to give the triazide structure the preference.

CHAS. A. ROULLER

An occurrence of fumaric acid and of inositol. L. ZECHMEISTER AND P. SZÉCSI. Lab. "Chinoïn" Fabr. chem.-pharm. Produkte A.-G., Budapest-Ujpest. *Ber.* 54B, 172-3(1921).—A 70% alc. percolate of 1 kg. freshly dried cassweed (*Capsella bursa pastoris*) was freed from alc. *in vacuo*, sepd. from oily deposits and exhausted with C_6H_6 . The brown sirup (465 g.) remaining on evap. the aq. soln. was thoroughly kneaded 3 times with EtOH, then with MeOH until a powder resulted. The MeOH soln. was concd. *in vacuo* to a thin sirup which after several days in the ice chest deposited 1.4 g. acid potassium fumarate, $2C_4H_2O_4K.C_6H_5O_6$, whetstone-shaped crystals, identical with the product obtained from the acid and the calcd. amt. of KOH. An aq. infusion of 1 kg. of the drug, pptd. successively with neutral and basic Pb acetate, freed from Pb with H_2S , concd. to a sirup *in vacuo* and thoroughly kneaded with alc. yielded, on letting the alc. ext. stand in the cold, 0.3 g. of inositol, m. 218-9°. C. A. R.

The chemistry of the glutamic acids. XII. The simultaneous occurrence of 1,2- and of 1,3-addition to "nascent" glutamic ester. CHRISTOPHER KELK INGOLD AND JOCELYN FIELD THORPE. Imp. Col. Sci. Tech., South Kensington. *J. Chem. Soc.* 119, 492-501(1921). In order to make use of the unsatd. modification of glutamic ester in the so-called "nascent" state, I. and T. generate this substance from from Et β -hydroxyglutarate in a hot alc. soln. of $CNCHNaCO_2Et$, which is also a reagent for unsatd. compds. and combination with the reagent occurs to an extent detd. by the velocity of the reaction in relation to that of the tautomeric change. 91% of the product was isolated and identified as follows: unchanged Et β -hydroxyglutarate; normal Et glutamate; Et ω -cyanomethanetriacetate, b_{11} 203-4°; ethyl cyclopentanone-2,4-dicarboxylate, $C_8H_{13}O_5$, long needles, m. 78-9°. The aq. alc. soln. gives an intense blue color with $FeCl_3$. Semicarbazone, long needles, m. 160°. On hydrolysis with 10 parts 8% H_2SO_4 , the ester yields cyclopentanone-3-carboxylic acid. Ethyl β -cyanomethylglutarate, $CNCH_2CH(CH_2CO_2Et)_2$, b_{15} 158-60°. Ethyl cyclopentanone-3-carboxylate, $C_8H_{13}O_5$, by hydrolysis of the above ester with concd. H_2SO_4 in the cold, b_{14} 120°. Et methanetriacetate, b_{14} 172-3°. C. J. WEST

Quantitative reduction by hydriodic acid of hydrogenated malonyl derivatives. I. The amides and sym-dialkyl- and aryl-substituted amides of mono- and dibromomalonic acids. JOHN VALENTINE BACKES, RALPH WINTON WEST, AND MARTHA ANNIE WHITELEY. Imp. Col. Sci. Tech. South Kensington. *J. Chem. Soc.* 119, 359-79(1921).—The amides and alkyl- and aryl-substituted amides and ureides of $BrCH(CO_2H)_2$ and $CBR_2(CO_2H)_2$ are quant. reduced by HI at the ordinary temp. to the corresponding deriv. of the acid. The reduction is carried out by adding solid KI to a soln. of the Br compd. in glacial AcOH; at temps above 60° the reaction is instantaneous, but requires about 2 hrs. at the ordinary temp. The course of the reaction may involve hydrolysis of the Br compd. with liberation of an active brominating agent, probably $HBrO$ or a deriv., which is immediately reduced by the HI. In the bromination of malonamide the reaction takes place in accordance with the following rules: the H atom in the β -position in each Ph group and 1 of the H atoms in the malonyl group are equally susceptible to Br and the 1st and least brominated deriv. is $CHBr(CONHC_6H_5)_2$. The 2nd H atom in the malonyl radical is more readily replaced than either of the H atoms in the α -positions in the Ph groups and the 2nd product is $CBR_2(CONHC_6H_5)_2$. Only 1 of the 2 H atoms in the α -positions in each Ph group can be replaced by direct bromination

and the final product is $\text{CBr}_2(\text{CONHC}_6\text{H}_4\text{Br})_2$. The constitution of these compds. is easily detd. since the Br of the malonyl complex is quant. replaced by H by the action of HI. *Bromomalonyl amide*, $\text{CHBr}(\text{CONH}_2)_2$, from 5 g. $\text{CH}_2(\text{CONH}_2)_2$ in 400 cc. glacial AcOH and 25 cc. of 10% (by vol.) Br in AcOH, slowly mixed with const. shaking, and spontaneous evapn. of the solvent, hard prisms from EtOH, m. 181° (decompn.). In the prepn. of $\text{CBr}_2(\text{CONH}_2)_2$, the yield can be increased to 94% by the addition of AcONa to neutralize the HBr formed in the reaction. It m. 203° without decompn. *Bromomalonyldimethylamide*, $\text{CHBr}(\text{CONHMe})_2$, by treating 8 g. $\text{CH}_2(\text{CONHMe})_2$ in 50 cc. CHCl_3 with 30 cc. 10% Br in CHCl_3 at $40-50^\circ$, thin prisms from EtOH, m. 164° . $\text{CBr}_2(\text{CONHMe})_2$ (Freund, *Ber.* 17, 782) is prepd. by the action of excess of Br on a hot soln. of $\text{CH}_2(\text{CONHMe})_2$ or its Br deriv. in CHCl_3 . $\text{CH}_2(\text{CONHMe})_2$ was prepd. in 95% yield by shaking a mixt. of 16 g. $\text{CH}_2(\text{CO}_2\text{Et})_2$, 30 g. 33% EtNH₂ and 0.2 g. NaOH for about 1 hr. at 0° . *Bromomalonyldiethylamide*, $\text{CHBr}(\text{CONHEt})_2$, thin prisms, m. 160° . *Dibromomalonyldiethylamide*, $\text{CBr}_2(\text{CONHEt})_2$, by the further bromination of the mono-Br deriv., in CHCl_3 , thin prismatic needles from EtOH-petrol. ether, m. 78° . *Malondipropylamide*, $\text{CH}_2(\text{CONHPr})_2$, by allowing 7 g. PrNH₂ and 9.6 g. $\text{CH}_2(\text{CO}_2\text{Et})_2$ to stand in a sealed tube for 24 hrs., and then heating 6 hrs. at $125-130^\circ$, 6-sided plates from acetone, m. 139° . *Bromomalondipropylamide*, $\text{CHBr}(\text{CONHPr})_2$, prismatic needles from dil. alc., m. 134.5° . *Dibromo derivative*, $\text{CBr}_2(\text{CONHPr})_2$, by brominating the amide in hot H₂O, or the Br deriv. in CHCl_3 , hexagonal prisms from light petroleum, m. 78.5° . *Malondibutylamide*, $\text{CH}_2(\text{CONHC}_4\text{H}_9)_2$, pearly plates from light petroleum, m. 132.5° . *Bromo derivative*, $\text{CHBr}(\text{CONHC}_4\text{H}_9)_2$, thin prisms, m. 103° . *Malondisobutylamide*, $\text{CH}_2(\text{CONHC}_4\text{H}_9)_2$, needles, m. 128.5° . *Bromo derivative*, $\text{CHBr}(\text{CONHC}_4\text{H}_9)_2$, stout prisms from C_6H_6 -petrol. ether, m. 151° . The quickly cooled soln. in petroleum forms a gel, which slowly becomes limpid and deposits crystals. *Dibromo derivative*, $\text{CBr}_2(\text{CONHC}_4\text{H}_9)_2$, by bromination in CCl_4 , thin prisms, m. 100° . *Malonyldibenzylamide*, $\text{CH}_2(\text{CONHCH}_2\text{Ph})_2$, by boiling 20 g. $\text{CH}_2(\text{CO}_2\text{Et})_2$ and 27 g. PhCH_2NH_2 for 20 hrs., plates, m. 142° . *Bromo derivative*, $\text{CHBr}(\text{CONHCH}_2\text{Ph})_2$, is the 1st product of bromination in AcOH, thin needles, m. 167° . In the prepn. of the *dibromo derivative*, the mixt. of the two compds. was dissolved in Br in hot CCl_4 and boiled until heavy prismatic crystals of the *dibromo derivative* sepd., hard, prismatic crystals, m. 128° . $\text{CH}_2(\text{CONHCO}_2\text{Et})_2$ is conveniently prepd. by warming 10 g. $\text{CH}_2(\text{CO}_2\text{H})_2$, 18 g. urethan and 16 cc. POCl_3 at 80° . *Bromo derivative*, prisms, from dil. alc., m. 148° . *Malonmonophenylamide* $\text{NH}_2\text{COCH}_2\text{CONHPh} \cdot 0.5\text{H}_2\text{O}$, obtained by heating 15 g. PbNH_2 and 36 g. $\text{CH}_2(\text{CO}_2\text{Et})_2$ for 6 hrs. at 120° and shaking the cold product with 2 vols. concd. NH_4OH for 2 hrs., pearly plates which lose H_2O at 100° and m. 164° . *Dibromo derivative*, $\text{NH}_2\text{COCHBrCONHC}_6\text{H}_4\text{Br}$, by the action of Br in AcOH upon the amide in AcOH, needles, m. 200° (decompn.). *Dibromomalonmonobromophenylamide*, $\text{NH}_2\text{COCBrCONHC}_6\text{H}_4\text{Br}$, by using 3.5 mols. Br in AcOH, stout prisms, m. 142° . *Dibromomalonmonodibromophenylamide*, $\text{NH}_2\text{COCBrCONHC}_6\text{H}_4\text{Br}_2$, by using a large excess of Br, prisms from dil. alc., m. 194° . *Bromomalon-p-bromoanilide* (A), $\text{CHBr}(\text{CONHC}_6\text{H}_4\text{Br})_2$, from 20 g. $\text{CH}_2(\text{CONHPh})_2$ in 250 cc. hot glacial AcOH and 135 cc. 10% Br in CHCl_3 , thin, silky needles, m. 236° . The constitution was detd. by direct bromination of $\text{CH}_2(\text{CONHC}_6\text{H}_4\text{Br-p})$ and by the formation of the same compd. by reduction of A with HI. *Dibromomalon-p-bromoanilide*, $\text{CBr}_2(\text{CONHC}_6\text{H}_4\text{Br})_2$, from 15 g. A in 1 l. boiling glacial AcOH and 30 cc. 10% Br in CHCl_3 ; after 50-60 hrs. the Br disappeared and the tri-Br deriv. formed a heavy glassy cryst. substance, m. 164° . *Dibromomalon-2,4-dibromoanilide*, by using 23.5 mols. Br in glacial AcOH, needles, m. 146° . When hydrolyzed with HCl, this yields 2,4,6- $\text{Br}_3\text{-C}_6\text{H}_2\text{NH}_2$. *Dibromomalon-2,4,6-tribromoanilide*, $\text{CBr}_2(\text{CONHC}_6\text{H}_2\text{Br}_3)_2$, by the action of a large excess of Br upon $\text{CH}_2(\text{CONHC}_6\text{H}_2\text{Br}_3)_2$, flat prisms, m. 174° . $\text{CBr}_2(\text{CONH-Ph})_2$, hard compact prisms, m. 147° . *Bromomalon-p-toluidide*, $\text{CHBr}(\text{CONHC}_6\text{H}_4)_2$.

thin plates, m. 211° , from $C_4H_5 \cdot EtOH$. *Dibromomalon-p-toluidide*, $CBBr_2(CONHC_6H_4)_2$, by heating the mono-Br deriv. with Br in $CHCl_3$, short prismatic needles from CCl_4 , m. 137° . *Dibromomalon-2-bromo-p-toluidide*, $CBBr_2(CONHC_6H_4Br)_2$, by heating the components in $CHCl_3$ until the solid had dissolved, short prisms from $EtOH$ or CCl_4 , m. 150° . Reduced with HI, *malon-2-bromo-p-toluidide* results, m. 180° , identical with that from $CH_3(CO_2Et)_2$ and $BrC_6H_4NH_2$. *Bromomalon-4-bromo-o-toluidide*, needles, m. 233° . Upon reduction with HI, *malon-4-bromo-o-toluidide*, m. 216° , is obtained. *Dibromomalon-4-bromo-o-toluidide*, thin prisms from CCl_4 , m. 137° . *5-Bromo-1,3-diphenylbarbituric acid*, $CO(NPhCO)_2CHBr$, prep'd. by adding 13 cc. 10% Br in $CHCl_3$ to 7 g. $CO(NPhCO)_2CH_2$ in 50 cc. $CHCl_3$, hard warts, m. 155° . The *o*-Br or 5,5-Br₂ derivs. of various barbituric acids are quant. reduced by HI. Typical chromoketones, such as $PhCOCH_2Br$, and α -bromo- α -nitrocampor, are also quant. reduced by HI.

C. J. WEST

The formation and properties of dithio ketones ($R_2C:S:S$) and dithio ethers ($RS:S$). I. KUVKJI GOSAI NAIK. Imperial Col. Sci. Tech., South Kensington. *J. Chem. Soc.* 119, 379-85 (1921).—The object of the present study is to show to what degree the instability of the dithio grouping is affected by the mol. condition of the remainder of the mol. and to compare the relative stability exhibited by corresponding dithio ketones of the general formula $R_2C:S:S$. The work is directly connected with "mustard gas" and the value of related compounds in chemical warfare. *Dithiomesoxanilide*, $(NHPhCO)_2C:S:S$, was prep'd. by boiling 9 g. $CH_2(CONHPh)_2$ and 10 g. S_2Cl_2 in 50 cc. C_6H_6 for 3 hrs.; from acetone it formed short needles, m. $214-5^{\circ}$. The reaction appears to take place in 1 stage only. HNO_3 gives a cryst. product, $C_{14}H_{10}O_4N_4S_2$, m. $232-3^{\circ}$. Upon hydrolysis with alkalis, $PhNH_2$ and an alkali salt are formed, which latter liberated H_2S on treatment with acid. Br gives $CHBr(CONHC_6H_4Br)_2$ (see preceding abstr.). Upon reduction, H_2S and $CH_2(CONHPh)_2$ were formed. *Dithiomesoxodimethylamide*, $(NHMeCO)_2C:S:S$, slender needles, m. $216-7^{\circ}$. *Dithiomesoxodibenzylamide*, $C_{17}H_{14}O_4N_4S_2$, fine crystals, m. 204° . *Malondimethylanilide disulfide*, $S_2S[CH(CONMePh)]_2$, m. $218-9^{\circ}$. *Methylmalonanilide disulfide*, $C_{22}H_{20}O_4N_4S_2$, cryst. powder, m. $229-30^{\circ}$. *5-Disulfido-1,3-diphenylbarbituric acid*, $C_{24}H_{18}O_4N_4S_2$, m. 181° .

C. J. WEST

Polysaccharides. II. The constitution of diamylose. P. KARRER AND C. NÄGELI. Zürich. *Helvetica Chim. Acta* 4, 169-73 (1921); cf. *C. A.* 15, 61 (1921).—Little is known as to the constitution of the two "basic amyloses," α -diamylose and β -triamylose, except that they give *d*-glucose on hydrolysis. Their compn. further indicates that in diamylose and triamylose, there is the anhydride of a di- and trisaccharide, resp. The identity of these saccharides has not been proved as yet and their inner structure has not been det'd. This is of fundamental importance in the problem of the structure of starch. α -Tetramylose in contact with $AcBr$ for 5 days at room temp. is quant. changed to acetohromomaltose. The process is carried out so that the tetramylose first forms the acetylated α -diamylose, whereupon the $AcBr$ opens up the anhydride ring. The acetohromomaltose was identified by transforming it into heptacetylmaltose, m. $177-8^{\circ}$, by using Ag_2CO_3 as suggested by Fischer. This m. p. is 1° higher than that found by Fischer. It was further identified by measuring its $[\alpha]$ in $C_2H_5Cl_4$. The heptacetylmaltose gave maltose on boiling with water, thus proving that α -diamylose is an anhydride of maltose. This is the first known anhydride of a disaccharide. The values of the $[\alpha]$ in $C_2H_5Cl_4$ and H_2O of the compds. studied are given. R. CHESTER ROBERTS

The Friedel-Crafts' reaction. II. Migration of halogen atoms in the benzene nucleus. MAURICE COPISAROW AND CYRIL N. H. LONG. Univ. Manchester. *J. Chem. Soc.* 119, 442-7 (1921); cf. *C. A.* 11, 1647. In the reaction of $AlCl_3$ upon $PhBr$, condensation, migration and pyrogenic-like fission of the C_6H_5 nucleus take place. These 3 distinctly different reactions form 3 successive stages, which overlap one another to

a considerable extent. Owing to the energetic character of the last 2 reactions, the ordinary condensation with the formation of $(\text{BrC}_6\text{H}_4)_2$ is greatly inhibited. The fission of the C_6H_4 nucleus with the formation of C, Br derivs. of C_{10}H_8 , $\text{C}_{14}\text{H}_{10}$, etc., is favored by extreme conditions of temp. and concn. of AlCl_3 . The migration of the Br atom, as estimated by the formation of C_6H_6 , could be increased to 83% of the theoretical by ensuring the continuous removal of the C_6H_6 and by carrying out the reaction in a current of HCl or H. PhOH, by fixing the Br, also facilitates the progress of migration, but only to a certain extent, owing to the great reactivity of PhOH with AlCl_3 . AlCl_3 and AlBr_3 have no such effect upon PhCl and PhBr, resp. Thus, in order to avoid side reactions in Friedel-Crafts' reactions, in which PhX are to be employed, Cl derivs. must be used with AlCl_3 .

C. J. WEST

The binary system, aniline-acetic acid. EDMUND ARTHUR O'CONNOR. Univ. Melbourne. *J. Chem. Soc.* 119, 400-403(1921).—No compd. corresponding to the formula $\text{PhNH}_2\cdot\text{AcOH}$, was found. The compound $\text{PhNH}_2\cdot 2\text{AcOH}$ m. 18.7° ; the compound $2\text{PhNH}_2\cdot\text{AcOH}$ m. -19.4° and is metastable under all conditions. These with pure AcOH and PhNH_2 were the only solid phases obtained. The invariant points where the compound $\text{PhNH}_2\cdot 2\text{AcOH}$ exists in equil. with the liquid phase and AcOH and PhNH_2 are at temps. of -2.4° and -15.6° , resp. The compound $2\text{PhNH}_2\cdot\text{AcOH}$ coexists with PhNH_2 and soln. at -19.8° .

C. J. WEST

Phenol losses in the decomposition of phenate liquors. B. HARDMAN. *Chem. Trade J.* 68, 561-2(1921).—During the war there was secured from one of the British PhOH plants a by-product which contained 4-5% PhOH as the Na salt. The salt was recovered by leaching, filtering and by an acid treatment in Ph-lined tanks, "striking vats," after the following manner. The clear soln. was decompd. in the vats with niter cake and if necessary with chamber acid. The temp. rose from 55° to 60° and the contents of the vats were cooled to 40° by agitation for 16 to 20 hrs. After standing for 4 hrs., the layers were removed and during the whole process of agitation and settling a current of air flowed constantly over the liquor. In this way SO_2 and steam were carried up the flue. Expts. were effected in the lab. to ascertain the amt. of PhOH lost up the flue and to find out what the effect of time and temp. is on these losses. A table is given showing what the losses are at a given temp. in certain time intervals, and it is shown that the proportionate losses at the given temps. have a remarkably close relation to the proportionate vapor pressures of H_2O compared at the same temp. and it seems quite possible that remarkable similarity between the proportionate losses of PhOH and the proportionate variations in the vapor pressure of H_2O at the same temp. may be utilized to plot a curve showing what losses of PhOH might be expected at any temp. during the same period of time. The expts. show that really enormous quantities of PhOH can be lost during "striking out." When the vents leading to the flues were closed during striking out and settling it was found that the amts. of PhOH expected were obtained. The results may be of interest to those using the CO_2 method of decomp. sodium carbolate solns.

H. E. WILLIAMS

The technical production of picric acid directly from benzene. J. PROCHÁZKA. *Chem. Listy* 15, 59-60(1921).—Polemical. The low yields obtained by Blechta (cf. *C. A.* 15, 753) show that the method is not economical. JOHN M. KRNO

The technical production of picric acid from benzene. FR. BLECHTA. *Chem. Listy* 15, 60(1921).—Polemical. Cf. preceding abstr. JOHN M. KRNO

Tautomerism of the phenols. III. Bisulfite and phloroglucinol. WALTER FUCHS. Deutsch. Tech. Hochschule Brünn. *Ber.* 54B, 245-9(1921); cf. *C. A.* 14, 3248.—Ten g. NaHCO_3 covered with 30 cc. H_2O are satd. with SO_2 , treated with 5 g. phloroglucinol (A), heated 80 hrs. in a H_2O bath, cooled somewhat, dild. with 4-5 vols. alc., shaken thoroughly, filtered, concd. to 0.5 the vol. of the original aq. soln., allowed to stand *in vacuo* for some hrs., drained as completely as possible, washed with concd. alc. and dried in

vacuo at 100°; there is thus obtained about 5 g. of a snow-white cryst. mass of the compn. A.NaHSO_3 , easily sol. in H_2O with neutral reaction, seps. from alc. in needles; in its aq. soln. SO_2 can be detected with I at most only in traces; it is very stable towards acids. It thus resembles very closely the phenol-bisulfites even to the extent of puffing up on heating like Pharaoh's serpents and leaving on Pt a gray stain which cannot be removed by ignition. Moreover, it is exceedingly stable towards KOH; only a minimal amt. of the SO_3 can be split off by allowing it to stand in aq. KOH for many hrs. and even on warming the alk. soln. on the H_2O bath; a considerable part, although by no means all, of the SO_3 can be split off, however, by the action of neutral BaCl_2 in the presence of H_2O_2 . Ba(OH)_2 at once produces a ppt. $\cdot \text{FeCl}_3$ (4%) gives an intense red-violet color (the soln. then contains Fe^{+} ions) destroyed by a drop of HCl , dil. KOAc or dil. NaHSO_3 and changed to an intense brown-green color by a drop of H_2O_2 ; addition of a drop of KOAc increases the intensity of this last color and imparts to it a tinge of red; very dil. H_2O_2 colors a soln. of the compd. after some time a faint red which becomes more intense on the H_2O bath. In another expt. in which 5 g. phloroglucinol was heated with the NaHSO_3 100 hrs. in a vigorously boiling H_2O bath there sepd. on cooling 9-10 g. of needles, analysis of which gave values corresponding approx. to a compd. A.3NaHSO_3 ; that some Na_2SO_4 was present is certain. The aq. soln. of the product gave no color with FeCl_3 and consumed but a minimal amt. of I; after treatment with KOH a large portion, but not all, of the added SO_3 could be detected. F. concludes that phloroglucinol forms with NaHSO_3 addition products entirely analogous in a number of characteristic properties to those previously obtained from phenols and NaHSO_3 , but it has not yet been detd. whether these products (especially the mono compd.) are acetal esters with the grouping $\cdot \text{CH}_2\text{C(OH)(OSO}_3\text{Na)}_2$ -, or hydroxysulfonates, $\cdot \text{CH}_2\text{C(OH)(SO}_3\text{Na)}_2$ -. IV. Replies. *Ibid* 249-52(1921).—Replies to Bucherer (C. A. 15, 81) and Herzog and Zeisel (C. A. 15, 86). CHAS. A. ROUVILLER

The hydrogenation of quinhydrone. EINAR BILMANN. *J. Soc. Leather Trades' Chemists* 5, 27-31(1921).—By dipping a piece of Pt foil in an acid soln. of quinhydrone, which is largely dissociated in aq. soln. into quinone and quinol, an electrode is formed which may be combined with an ordinary H electrode to form an element $[\text{Pt} | \text{C}_6\text{H}_4\text{O}_2, \text{C}_6\text{H}_4(\text{OH})_2 + 0.1 \text{ N HCl} | 0.1 \text{ N HCl} | \text{Pt}]$. In the reactions of this element 2 H ions disappear in the quinhydrone electrode and 1 mol. of $\text{C}_6\text{H}_4\text{O}_2$ is hydrogenated to $\text{C}_6\text{H}_4(\text{OH})_2$. The H-ion concn. of the electrode is proportional to the concn. of $\text{C}_6\text{H}_4(\text{OH})_2$ and inversely proportional to the concn. of $\text{C}_6\text{H}_4\text{O}_2$ and the potential of the element is independent of the abs. concn. The potential of such an element, with an equal H-ion concn. at both electrodes is at 18° 0.7044 v. and at 25° 0.6990 v. and the H pressure of the quinhydrone electrode is at 18° $10^{-24.4}$ atm. and at 25° $10^{-23.4}$ atm. In the construction of the electrode the vessels used were those ordinarily employed for the calomel electrode. The electrodes were of bright Pt foil 30×7 mm. The quinhydrone soln. used (usually 0.005 M) was made by dissolving in the acid soln. to be used or in dil. acid which was afterwards brought to the desired concn. The constancy of the electrode was tested with a soln. which was 0.005 M with regard to quinhydrone and 0.05 N with regard to H_2SO_4 . This was combined with a 0.1 N calomel electrode by means of 3.5 N KCl. In most tests the deviations rarely exceeded 0.2 and never 0.4 millivolts. Expts. with 0.1 N HCl gave more exact results, the greatest deviation being 0.07 millivolt. A series of elements was made with the following solns.: 0.1 N HCl, $[\text{H}^+] = 10^{-1.224}$; 0.1 N H_2SO_4 , $[\text{H}^+] = 10^{-1.171}$; 0.02 N H_2SO_4 , $[\text{H}^+] = 10^{-1.722}$; 0.1 N secondary citrate, $[\text{H}^+] = 10^{-4.284}$; mixts. of equal mols. M/15 KH_2PO_4 and M/15 Na_2HPO_4 , $[\text{H}^+] = 10^{-4.212}$; Sørensen's borate soln., $[\text{H}^+] = 10^{-9.741}$. The borate soln. gave no const. potential, being too basic, but the rest all showed the same potential against H electrodes of the same acidity, though the citrate soln. was less const. than the rest. It was found that H electrodes can be formed with toluquin-

hydrone and xyloquinhydrone, and an electrode was made with equal mols. of dialuric acid and alloxan. Also in *Ann. chim.* **15**, 109–57(1921).

G. W. STRATTON

Chemical constitution and rotatory power. V. Some derivatives of α -anisylethylamine. MARIO BETTI and ASSUNTA CAPACCIOLI. Univ. Siena. *Gazz. chim. ital.* **50**, II, 276–80(1920).—The results obtained from a polarimetric study of the aldehydeamino derivs. of β -naphtholbenzylamine were published previously (*C. A.* **3**, 73; **10**, 2890, 2891) and gave rise to an examn. of the rotatory power of other compds. of analogous structure. It was for this purpose that optically active α -aminophenylacetic acid and phenylethylamine (*C. A.* **6**, 2417) were prepd. The results here described relate to aldehyde derivs. of α -anisylethylamine (A), $\text{CH}_3\text{O.C}_6\text{H}_4\text{CHMeNH}_2$. This base was decompd. into its optical antipodes by the method described previously (*C. A.* **6**, 2417) and the *d*-form, $[\alpha]_D$ 22.68°, was obtained pure. Pure C_6H_6 was used as the solvent and 0.02 g.-mol. per 100 cc. of the substance was used. The EtOH soln. of the aldehyde and A were mixed and the product sought send. The following derivatives are described: *benzaldehyde*, yellowish oil which could not be crystd.; *salicylidene*, silky yellow needles, m. 66°, $[\alpha]_D$ —139.76°; *5-bromosalicylic aldehyde*, yellowish needles, m. 116–7°, $[\alpha]_D$ 34.73°; *p-dimethylaminobenzal*, yellow needles, m. 105°, $[\alpha]_D$ —170.21°; *vanillidene*, white needles, m. 130–1°, $[\alpha]$ not detd. because of insoly. in C_6H_6 ; *p-hydroxybenzal*, brown crystals, m. 162–3°, insol. in C_6H_6 ; *5-nitrosalicylidene*, yellow needles, m. 106–7°, $[\alpha]_D$ —4.00°. *o*- and *m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CHO}$, *o*- and *p*- $\text{HOC}_6\text{H}_4\text{CHO}$, vanillic, trimethylphloroglucinic and phthalic aldehyde gave noncryst. products with hydroxynaphthylbenzylamine. The results confirm what was observed before with other compds., viz. that the more acid the C_6H_4 nucleus becomes by the substitution of groups the less the *l*-rotation of the complex formed with the *d*-form of A.

E. J. WITZEMANN

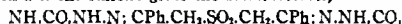
Chemical constitution and rotatory power. VI. Aldehydeamino derivatives of α -naphthylphenylaminomethane. SERGIO BERLINGOZZI. Univ. Siena, *Gazz. chim. ital.* **50**, II, 281–4(1920).—In a recent paper concerning the sepn. of α -naphthylphenylaminomethane (A) (*C. A.* **15**, 837) into its optical antipodes B, mentioned the analogy of this base to α - β -hydroxynaphthylphenylaminomethane (B) studied by Betti (*C. A.* **1**, 727). Betti found regularity in the variation of the rotatory power with aldehydeamino derivs. of this base and the data obtained are reviewed here. The results obtained with A conform with this earlier work. These aldehydeamino derivs. of A were prepd. by the action of equimol. amts. of the base and A in EtOH and heating on the H_2O bath for some mins. Both the *l*- and *d*-forms of A were used. The *l*-form gives a *p-dimethylaminobenzaldehyde* derivative, as shining scales, pale straw-yellow, m. 198–9°, $[\alpha]_D$ 222.62°. The *d*-form gives an *anisic aldehyde* derivative as white needles, 124–5°, $[\alpha]_D$ —180.92°. The *d*-form gives a *benzaldehyde* derivative as white prisms, m. 107–8°, $[\alpha]_D$ —162.94°. The *l*-form gives a *p-chlorobenzaldehyde* derivative as a white microcryst. powder, m. 110–11°, $[\alpha]_D$ 138.97°. The *l*-form gave an *o-chlorobenzaldehyde* derivative as white scales, m. 124°, $[\alpha]_D$ 130.37°. The *l*-form gave an *o-nitrobenzaldehyde* derivative as citron-yellow needles or scales, m. 100–1°, $[\alpha]_D$ 72.13°. The *l*-form gives a *3-nitrosalicylaldehyde* derivative as orange-yellow silky needles, m. 157–8°, $[\alpha]_D$ 171.89°.

E. J. WITZEMANN

Stereoisomeric derivatives of phenacyl sulfide. EMIL FROMM and ALFRED EHRHARDT. Univ. Freiburg i. B. *Ber.* **54B**, 187–93(1921); cf. *C. A.* **7**, 3745.—*Phenacyl sulfide monohydrazone anhydride*, $\text{N:CPh.CH}_2\text{S.GH}_2\text{CPh:N}$, from the sulfide boiled

a long time under a reflux with excess of $\text{N}_2\text{H}_4\text{H}_2\text{O}$ (A) in AcOH, crystals from AcOH or alc., m. 175°, also obtained by shorter or longer boiling of the 2 hydrazones below with AcOH. *Phenacyl sulfide syn-monohydrazone*, $\text{PhC(:NNH}_2\text{)CH}_2\text{SCH}_2\text{COPh}$, from the anhydride boiled a long time with dil. alc., m. 225°, regenerates the anhydride on short boiling with AcOH. *anti-Monohydrazone*, from equal mols. of the sulfide and A

in boiling alc., or without solvent, m. 128°, converted into the *syn*-compd. by short and into the anhydride by long boiling with AcOH. *Phenacyl sulfone monohydrazone anhydride*, m. 206°; *syn-hydrazone*, m. 185°, gives with BzH not the expected benzal deriv. but the anhydride; *anti-hydrazone*, m. 158°, gives on boiling with BzH the *anti-benzalhydrazone*, crystals from C₆H₆, m. 150°. *syn-Dihydrazone*, from the sulfone and 2 equivs. A slightly acidified with AcOH and heated a short time under a reflux or from the monohydrazones heated in faintly acid soln. with A, m. 156°; *debensal derivative*, yellow crystals from C₆H₆, m. 178°. When the sulfone is boiled in alc. with 2 equivs. A 2 substances are formed: the more easily sol. *anti-dihydrazone*, m. 172°, obtained exclusively from the *anti*-monohydrazone boiled with A in alc. (*dibenzal derivative*, yellow crystals from C₆H₆, m. 194°), and the less sol. *amphi-dihydrazone*, m. 188°, formed exclusively from the *syn*-monohydrazone and A in alc. (*dibenzal derivative*, yellow crystals from C₆H₆, m. 171°). *Phenacyl sulfide monosemicarbazone*, from equimol. amts. of the sulfide and H₂NCONHNH₂·HCl (B) in boiling alc., m. 237°. *Disemicarbazone*, obtained with excess of B in alc., m. 232°. *Phenacyl sulfone monosemicarbazone*, m. 258°. With excess of B in alc. the sulfone gives the *diaminobiurel*,

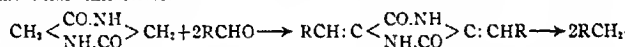


m. 252°. *Carbohydrazone*, CO.NH.N: CPh.CH₂SO₂CH₂CPh:N.NH, from the sulfone

heated in alc. with free H₂NCONHNH₂, m. 250°.

C. A. ROUILLER

Condensation of glycine anhydride with aldehydes. A new synthesis of *dl*-phenylalanine and *dl*-tyrosine. TAKAOKI SASAKI. Sasaki-Lab., Tokio. *Ber.* 54B, 163-8 (1921).—Glycine anhydride (A) condenses with aldehydes to 3,6-dialkylidene-2,5-diketopiperazines which can be converted by reduction into the anhydrides of the corresponding NH₂ acids and then, or practically simultaneously, by hydrolytic cleavage into the acids themselves:



The method is of great use in the synthesis of NH₂ acids containing a nucleus sensitive to hydrolytic agents. As the yields are excellent, it is believed the method is capable of wide application. Thus, 11.4 g. A heated 8 hrs. at 120-30° with 26.5 g. BzH, 33 g. anhydrous NaOAc and 51 g. Ac₂O, digested with warm H₂O after cooling and extd. with warm alc. gives 18 g. 3, 6-dibenzal-2, 5-diketopiperazine, yellowish scales from AcOH, decomp. 298-300°; 7.3 g. boiled 8 hrs. under a reflux with 50 cc. HI (d. 1.7) and 5 g. red P yields 8.4 g. crude and 6.9 g. pure *dl*-PhCH₂CH(NH₂)-CO₂H, leaflets from H₂O, decomp. 263-4°, while 2 g. in 120 cc. boiling AcOH slowly treated with 5 g. Zn dust and boiled 12 hrs. gives 2 g. of the *dibenzyl* compound (*dl*-phenylalanine anhydride), m. 290-1°. 3, 6-Dianisal-2, 5-diketopiperazine (9.5 g. from 5.7 g. A, 17 g. MeOC₆H₄CHO, 16.5 g. NaOAc and 25 g. Ac₂O heated 6 hrs. at 120-30°), decomp. above 300°; 5 g. boiled 3 hrs. with 50 cc. HI and 5 g. red P, treated with 6 g. fresh P and boiled 5 hrs. longer gave 4.5 g. *dl*-tyrosine. 3,6-Bis-*p*-acetoxybenzal-2,5-diketopiperazine (12.5 g. from 5.7 g. A, 15.3 g. HOC₆H₄CHO, 15 g. NaOAc and 30 g. Ac₂O heated 6 hrs. at 120-30°), yellow crystals, decomp. above 300°; 3 g. boiled 8 hrs with 40 cc. HI and 4 g. red P gives 2.5 g. *dl*-tyrosine.

CHAS. A. ROUILLER

Condensation of some dipeptide anhydrides with benzaldehyde. TAKAOKI SASAKI AND TOKUDJI HASHIMOTO. Sasaki-Lab., Tokio. *Ber.* 54B, 168-71 (1921); cf. preceding abstr.—*dl*-Alanylglycine anhydride (4.3 g.) heated 8 hrs. at 120-30° with 8.8 g. BzH, 10 g. NaOAc and 17 cc. Ac₂O gives 5 g. 1-acetyl-3-benzal-6-methyl-, 5-diketopiperazine, crystals from dil. alc., m. 163-4°. From 4.3 g. *dl*-leucylglycine anhydride, 6.6 g. BzH, 7.5 g. NaOAc and 13 cc. Ac₂O heated 8 hrs. at 120-30° is obtained 4.1 g. 1-acetyl-3-benzal-6-isobutyl-, 5-diketopiperazine, m. 152-3°. 6-*p*-Acetoxybenzyl compound (11

g. from 0.15 g. glycyl-L-tyrosine anhydride, 1.5 g. BzH, 3.4 g. NaOAc and 4 cc. Ac_2O), crystals from 50% alc., m. 153–4°, is optically inactive. CHAS. A. ROULLER

The synthesis of *dl*-3,4-dihydroxyphenylalanine. K. HIRAI. *Biochem. Z.* 114, 67–70(1921).—5 g. of dry finely powdered glycine anhydride are heated for 6 hours at 160–70°, with 16.6 g. vanillin, 14.3 g. H_2O -free NaOAc and 22.3 g. Ac_2O . The yellow reaction mixt. is first digested with hot H_2O and then with alc.; there is obtained a yellowish cryst. substance which is dried in a vacuum desiccator, giving 79.2% of almost pure di-[3-acetoxy-4-methoxybenzyl]glycine anhydride, hardly sol. in H_2O , more so in hot glacial AcOH. Recrystn. from boiling glacial AcOH yields slightly yellow crystals which do not melt below 280°. 4.1 g. of the condensation product were heated for 8 hrs. with 18 cc. of HI (d. 1.7) and 2 g. red P, with a reflux condenser, filtered from the P, acidified with AcOH and treated with $\text{Pb}(\text{OAc})_2$. The PbI was removed by washing with H_2O and the clear colorless filtrate treated with NH_4OH to ppt. the Pb compd. of dihydroxyphenylalanine. This was suspended in a little H_2O and decompd. with H_2S , which in turn is driven out by CO_2 . The filtrate from the PbS was quickly acidified with a little H_2SO_4 and evapd. under diminished pressure in a H atm. The residue when taken up with as little H_2O as possible, sepd. and dried in a vacuum desiccator over CaCl_2 yielded 64.7% of *dl*-3,4-dihydroxyphenylalanine, crystals m. 271–2°. F. S. HAMMETT

Catalytic hydrogenations with copper. PAUL SABATIER AND BENNOSUKE KUBOTA. *Compt. rend.* 172, 733–6(1921).—The catalyst was prepd. by reducing black "tetracupric hydrate" slowly with H_2 at 200°. The hydrogenations, carried out at 350°, a higher temp. than is necessary when Ni is used, gave in part different results from those obtained with Ni. PhCHO gave CO, PhH, and PhMe, but no CH_4 . PhAc and PhCOEt were slowly but smoothly reduced to PhEt and PhPr. BzCH_2Ac was split primarily to PhAc and AcH, EtOH and PhEt appearing as secondary products. Quinone formed chiefly hydroquinone. $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$ gave PhH, CO, and H_2O , along with considerable phthalide, part of which in turn gave PhMe and CO. BEN H. NICOLET

Method of catalytic hydrogenation. JEAN NIVIERE. *Bull. soc. chim.* 29, 217–9(1920).—A very simple method, used by the author "in the terpene series", is described. Details are given for PhCHO , PhNO_2 , and benzalacetone. The reaction is carried out in the presence of 0.1–2 g. PtCl_2 or PdCl_2 in a heavy bottle. The H_2 is furnished by CaH_2 ("hydrolite," 1 g. = 1 liter of H_2) which is introduced in an inverted specimen tube sealed to the end of a glass rod passing through the rubber stopper. If more than one charge is required, fresh CaH_2 is added when heat is no longer evolved. Excellent results are claimed, and the method is said to be suited to "the most rudimentary laboratory." BEN H. NICOLET

Hydrogenation of aromatic compounds with the aid of platinum. IV. Dependence of the catalytic hydrogenation on the presence of oxygen. RICHARD WILLSTÄTTER AND ERNST WALDSCHMIDT-LEITZ. Bayer Akad. Wiss. München. *Ber.* 54B, 113–38(1921); cf. C. A. 13, 577.—The expts. of W. and W.-L. show that the intermediate products in catalytic hydrogenation with metals are not hydrides of the metals but that they contain both O and H. Oxidized Pt and Pd contain the H which they take up in a more easily dissociated union, in a more reactive form, than Pd or Pt hydride; when the H is dissociated away, the compd. of the metal containing active O is regenerated. It is, therefore, probable that the oxidized metal, the first intermediate product, reacts with the H in such a way that the second intermediate product in the hydrogenation is at the same time a superoxide (or oxide) and a hydride. The assumption that H and active O are chemically bound to a Pt atom means either that the second intermediate product is formed by the splitting off of H_2O and contains bivalent Pt or, which is more probable, that it is formed by addition and contains quadrivalent Pt, the transport of the H, therefore, depending upon a play between two valency stages of the Pt,

thus: $\text{Pt} + \text{O}_2 = \text{Pt.O.O}$; $\text{Pt.O.O} + \text{H}_2\text{O} = \text{Pt(OH)OOH}$; $\text{Pt.O.O} + \text{AcOH} = \text{Pt(OAc)-OOH}$; Pt.O.O (or the hydrate or acetate) $+ \text{H}_2 = \text{H}_2\text{Pt.O.O}$ (or the hydrate or acetate).

An improvement in the method of prep. the Pt sponge (*C. A.* 6, 2613) consists in using KOH instead of NaOH; as the result of the formation of the difficultly sol. K_2PtCl_4 the reduction proceeds more slowly, so that sudden rises in temp., foaming and mirror formation are more easily avoided and the Pt seps. in very satisfactory form; 80 cc. of a H_2PtCl_4 soln. (containing some HCl) from 20 g. Pt and 150 cc. of 33% HCHO at -10° are treated dropwise, with vigorous stirring, with 420 g. of 50% KOH (keeping the temp. below $4-6^\circ$), then warmed 0.5 hr. at $55-60^\circ$, washed by decantation until alkali- and Cl-free, gently drained (keeping the Pt always covered with H_2O), quickly pressed between filter paper, and evacuated about 10 hrs. with a high-vacuum pump. It reaches constancy in wt. in 1-2 days and now no longer forms H_2O on standing in a closed bottle. The almost deoxygenated prep. very easily absorbs O again and changes into efficient sponge, containing active O, which is stable indefinitely. To recharge Pt which has become inactive in the hydrogenation app., the shaking bottle is evacuated, then air or O is admitted with shaking and the bottle is again evacuated a short time before the H is admitted. During the H transportation the Pt is finely divided and powdery, especially on charging with O, but when the absorption of the H ceases or slows up the Pt settles generally in thick flocks; the change in appearance is marked when in the course of the hydrogenation the O content of the Pt is used up. Pd sponge also always contains O in active form; it is prep. by treating 100-50 cc. of a HCl soln. of 4.25 g. Pd and 50 cc. of 33% HCHO at -10° with 100 g. of 50% KOH in the course of 10 min. (the temp. fluctuates around $0-3^\circ$) and warming 15 min. at 60° . It had previously been found that in the hydrogenation of C_6H_6 it made practically no difference whether the Pt had or had not previously been charged with O. W. and W.-L. now find that 4 hrs. shaking of the Pt in AcOH with H does not weaken its activity in the hydrogenation of menthene but does so greatly in the hydrogenation of C_4H_8 ; *e. g.*, while the absorption of H in the presence of Pt so treated was 15 cc. in 15 min., it rose to 190 cc. after activation of the Pt with O. This apparent difference in the behavior of the olefin and the aromatic hydrocarbon, however, is due only to the difference in velocity of the two hydrogenations, that of the olefin being so easy that a very minute amt. of O in the Pt suffices to bring it about. The Pt can be completely deoxygenated by shaking with H 30 hrs. at room temp. or 8 hrs. at $50-60^\circ$; it then no longer dissolves in HCl and liberates no I from acidified KI soln. Such a Pt is incapable of effecting any hydrogenation whatever, either of C_6H_6 , an olefin or a diolefin, but treatment with O completely restores its activity. In all hydrogenations, just as when it is shaken alone in AcOH with H, the Pt loses its O; in those hydrogenations proceeding with moderate (C_6H_6) or small velocity (phthalic acid) the velocity falls to zero if a relatively small amt. of catalyst is used and the latter no longer reacts with acidified KI soln. There are many Pt poisons, like easily oxidized org. compds. containing O and S compds., whose toxic action may be overcome by increased admission of O or by increasing the amt. of the catalyst. A typical poisoning of this kind is produced by glycerol and, to a smaller extent, by thiophene. In the case of aromatic compds. which are hydrogenated more slowly than C_6H_6 were observed poisonings which could be overcome only by increasing the amt. of catalyst enormously and initiating the hydrogenation by adding C_6H_6 . Although 30 min. evacuation of the Pt at room temp. does not diminish its activity in the hydrogenation of C_6H_6 8 hrs. continuous evacuation with a high-vacuum pump markedly diminishes and 3 hrs. evacuation at 100° almost completely destroys its activity. Completely deoxygenated Pd sponge is likewise incapable of effecting hydrogenation even of diolefins. Colloidal Pd, like the sponge, is also

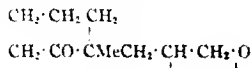
inactive when O-free. The lower oxides of Ni produced by heating NiO with H at 200° and 230–300° are capable of catalyzing hydrogenations, but such is not true of elementary Ni, even when prepd. with the greatest care at 350°; this, however, is activated by O, of which such a minute amt. is necessary that the activated product does not markedly react with acidified KI. The observations which have been made on the role of O lead to the following improvements in hydrogenations with Pt sponge: decrease in the amt. of catalyst, avoiding decreases in velocity by fatigue of the catalyst, overcoming disturbances produced by impurities, making it possible to compare velocities of hydrogenation. Pd sponge offers no advantages over Pt sponge and on account of its great power to absorb H it is more difficult to activate with O and the process is dangerous or impracticable. For an ordinary charge of substance to be hydrogenated (5–10 g.), 0.1–0.2 g. Pt is used, which with olefins is rarely activated, with aromatic compounds after some hrs. or every hr. by shaking a short time with air; difficultly hydrogenated substances are reduced at 60°. It is not necessary to use any considerable excess pressure of H. The velocity of catalytic hydrogenations with Pt sponge depends, not on a diffusion process as believed by Böeseken, but on the true hydrogenation velocity and the decrease in the amt. of the catalyst, as the oxygenated black or colloid, while transporting the H to the substance reduced, is at the same time reduced to the inactive hydride. As the loss of O by the catalyst often appears to proceed with uniform velocity, the reactivity of unsatd. compds. can be compared by the amts. of H which can be transported to them by a given amt. of oxygenated catalyst until it is exhausted. Thus, 0.1 g. Pt transported at 20° 1.2 l. H to C_6H_6 , about 9 l. to tetrahydrobenzene, 30 cc. to $C_6H_5(CO)_2O$ and 40 cc. to pyrrole. Another measure leading to similar comparative values is the initial velocity produced by the oxygenated catalyst; 0.1 g. Pt at 20° transported 80 cc. H to C_6H_6 in the first 15 min. and 130 cc. in 30 min., and 500 and 970 cc., resp., to tetrahydrobenzene. Further investigation is necessary to det. the influence of temp. and of the relative amts. of catalyst. Also the substance to be reduced (or its solvent) may be itself an O acceptor and, together with the H and more rapidly than the latter, bring about the deoxygenation of the catalyst, thereby giving too low hydrogenation velocities. Glacial AcOH had previously been found to be the most suitable solvent for hydrogenations with Pt sponge; it has now been found that H dissolves to about the same extent in AcOH and in C_6H_6 ; so the greater effectiveness of AcOH cannot be ascribed to the greater soly. of H in it. C. A. R.

Molecular coefficient of refraction, its additive behavior and its applicability to the determination of constitution. III. **The numerical relationships in the series of the polymethylene compounds.** FRITZ EISENLOHR, Albertus-Univ., Königsberg i. Pr. *Ber.* 54B, 290–320 (1921); cf. *C. A.* 15, 1307.—The relationships between the values for $M \times n_D^{20}$ for the polymethylenes are not always as unambiguous as for the monocyclic aromatic hydrocarbons. In the first place, the Sabatier hydrogenation method on the one hand and those of Willstätter and Skita on the other yield different products which are in part the *cis*- or *trans*-isomers in pure or, rather, approx. pure form but are also in part mixts. of the two forms. The data in the literature on the indices of refraction refer almost exclusively to the reduction products obtained by the Sabatier method, which consist predominantly of the *trans*-forms. The relationships discussed in the present paper refer to the *trans*-forms only. The laws established for the aromatic series cannot be applied directly to the polymethylene compds. While in the former it is the number rather than the positions of the side chains which is the detg. factor in the deviation, E , of the value of $M \times n_D^{20}$ from the calcd. value, in the polymethylenes the influence of the number of substituents, as compared with that of their position, practically disappears. *Cycloparaffins.*—Taking into consideration the optical influence of ring formation (—4.2 units for a 5-membered, —3.4 for a 6-membered ring), the influence of the introduction of a side chain (unlike the C_6H_6 hydrocarbons, this influence is exerted

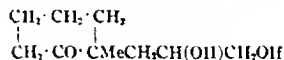
in the first member of the homologous series, *i. e.*, when the side chain is Me) is numerically equal to -0.73 and -0.88 unit for cyclopentane and cyclohexane, resp. For cyclohexane the introduction of two or more Me groups in the positions indicated by the numbers in parentheses produces the following changes in the value of $M \times \pi_D^{20}$: (1, 1) -0.77 ; (1, 2) -0.61 ; (1, 3) -1.29 ; (1, 4) -1.68 ; (1, 3, 4) -1.25 ; (1, 3, 5) -1.61 ; (1, 2, 4, 5) -1.19 . The depression for the *p*- (or *sym*-) derivs. is $0.5 \times$ no. of substituents greater than that for the *o*- (or *vic*-) compds. and the values for the *m*- (or *asym*-) forms are to those for the *p*- (or *sym*-) isomers as $0.7:1$. *Cycloolefins*.—An ethylene linking in a 6-membered ring (cyclohexene) produces only the usual decrement in $M \times \pi_D$; the same is true of cyclohexamethene and of the other compds. of the 5-, 6- and 7-membered rings but holds for the semicyclic compds. only as long as the H atoms of the methene group are not substituted. For endo- as well as semicyclic unsatd. polymethylenes, $M \times \pi_D^{20}$ varies widely according as the side chains are attached to the doubly bound C atoms in the ring, near this position or far from it. Especially marked is the difference between derivs. with substituents on both unsatd. C atoms, which show a high exaltation, and those with substituents far from the ethylene union, which show a considerable decrement. The semi- shows no peculiarity as compared with the endocyclic ethylene union as long as the substituents are on the ring. The introduction of a Me group does not seem to exert the same influence in a 5- as in a 6-membered unsatd. ring and the nature of the substituent also influences the value of E (0.03, 0.66, 1.12 and 1.24 for Me, Et, Pr and iso-Pr, resp., in position 1 in cyclohexene). In the semicyclic compds. the substitution of Me or Et for one of the H atoms of the methene group produces an exaltation of about 1.15, the substitution of both H atoms an exaltation of about 2.15 in the 6-membered ring series; in the 5-membered ring series the values are 0.82 and 1.65, resp. *Alcohols and ketones of the polymethylenes*.—Calling the value of E for cyclohexane 0, that for cyclohexanol and cyclohexanone is 0.34 and 0.35, resp.; if, now, 0.35 is subtracted from the observed values for the substituted alcs. and ketones the resulting values are almost identical with those obtained for the corresponding substituted hydrocarbons. In conclusion, it may be said that the values E , obtained by comparing the observed values of the mol. coeff. of refraction with those calcd. from the constns. for individual atoms and union equivs., afford an invaluable aid in the detn. of mol. structure and as a criterion of purity.

CHAS. A. ROULLER

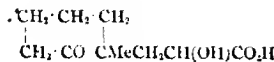
The oxidation of α, α -methylallylcyclohexanone with alkaline permanganate. R. CORNUBERT. *Compt. rend.* 172, 982-4 (1921); cf. *C. A.* 15, 1021.—Three % KMnO_4 in the presence of alc. KOH used to oxidize α, α -methylallylcyclohexanone (A) gave the following: (1) a liquid with a strong musty odor, $\text{C}_{10}\text{H}_{18}\text{O}_2$, b_D^{20} 128-31°, corresponding to I; (2) a considerable quantity of a colorless resin b_D^{20} 170-5°, whose mol. wt. was $(\text{C}_{10}\text{H}_{16}\text{O}_2)_n$; (3) a small quantity of crystals of II, m. 99.5-100°; (4) an acid product represented either by III or IV, but its exact constitution could not be detd.



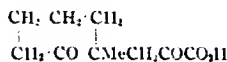
I



II



III



IV

The oxidation was carried out again, with soda lime instead of alc. KOH, giving modified results as follows: (1) a large quantity of a very viscous liquid of formula II, b_D^{20} 185-8°, which gave a benzoate, m. 141-2°; (2) a small quantity of crystals, m. 98-9°, of II, which were proved to be the same as those obtained above by a comparison of

their Bz derivs.; (3) a small quantity of a product b₂ 260–80° under 21 mm. which is the ether of the glycol II; (4) an acid product similar to that obtained with alc. KOH. A oxidized by KMnO₄ in the presence of soda lime behaves the same as allyldimethylacetophenone with KMnO₄ and alc. KOH. In contrast A oxidized in the presence of alc. KOH gives abnormal results.

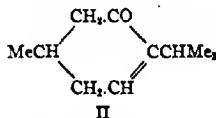
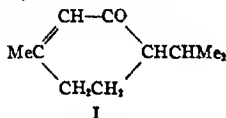
R. CHESTER ROBERTS

Synthesis of dimethyl cyclohexane-1,4-dione-2,3-dicarboxylate, an α -isomer of succinylsuccinic ester. BURCKHARDT HELFERICH. Univ. Berlin. *Ber.* 54B, 155–62 (1921).—1,4,2,3-C₆H₄(OH)₂(CN)₂ (Thiele and Meisenheimer, *Ber.* 33, 675(1900)), brown leaflets from H₂O, is obtained in 16 g. yield from 33 g. quinone dissolved in 500 cc. boiling H₂O and 170 cc. 5 N H₂SO₄, then treated as rapidly as possible with a concd. aq. soln. of 82 g. KCN, acidified with 170 cc. of 5 N H₂SO₄ and cooled as quickly as possible to 0°; 110 g. boiled 20 min. with 1320 g. of 50% KOH, cooled in ice, slowly poured into 2 l. ice-cold 5 N H₂SO₄, filtered after standing some time at 0° from the K₂SO₄ and exhaustively extd. with Et₂O gives 106 g. 3,6,1,2-C₆H₂(OH)₂(CO₂H)₂; 108 g. of this, dehydrated at 100°, boiled 30 hrs. in 900 cc. abs. MeOH and 48 cc. concd. H₂SO₄, neutralized while still warm with pptd. CaCO₃, dild. with an equal vol. of Et₂O, filtered and evapd. *in vacuo* to dryness gives 80 g. of *dimethyl 3,6-dihydroxyphthalate*, broad needles from H₂O, m. 141–2°, sol. in alkali with yellow color, in Et₂O with very faint bluish fluorescence, gives in H₂O a deep blue color with FeCl₃; *sodium salt*, from a concd. alc. soln. of the ester with NaOEt and much Et₂O, yellow amorphous ppt. remaining dry and crumbly in a desiccator but gradually deliquescent in the air; *dimethyl ether*, from 2 g. of the ester in 5 cc. MeOH and a slight excess of NaOMe allowed to stand 1 day with 3.5 g. MeI, short prisms from MeOH, m. 102–3°, gives no color with FeCl₃. *Diethyl ester*, m. 89°. From 10 g. of the Me ester added to 50 cc. of 2 N NaOH (previously cooled to the f. p.), then at once treated with 100 g. of 2.5% Na-Hg, shaken vigorously in the cold in a closed bottle, poured from the Hg into 87 cc. of iced 5 N H₂SO₄ under Et₂O, filtered and evapd. is obtained about 8 g. of *dimethyl cyclohexane-1,4-dione-2,3-dicarboxylate* (or the diol), short prisms from alc., m. 55–7°, gives in H₂O or alc. with FeCl₃ a deep red color, sol. in aq. alkalies and repptd. by CO₂ as an oil which solidifies on rubbing and seeding, gradually darkens in alk. soln. in the air, quickly reduces Fehling soln. and NH₃-AgNO₃ at room temp., gives in MeOH with NaOMe an almost white *sodium salt* which oxidizes very easily in the air. *Disemicarbazone*, needles from alc., m. 177–8° (decompn.). The ester (1 g.) is hydrolyzed by boiling 0.5 hr. with 4 cc. H₂SO₄ and 15 cc. H₂O to cyclohexane-1,4-dione. The ester (8.7 g.) allowed to stand 1 hr. in 24 cc. MeOH with 14 g. MeI and 1.8 g. Na in 25 cc. MeOH in a tightly closed bottle, then quickly evapd. to dryness *in vacuo*, taken up in H₂O and acidified with 16 cc. of 5 N H₂SO₄ gives almost quant. *dimethyl 3-methylcyclohexane-1,4-dione-2,3-dicarboxylate*, sinters 89°, m. 92°, gives a deep red color with aq. FeCl₃, dissolves without color in alkalies and is repptd. by CO₂; its alk. soln. reduces Fehling soln. on boiling, NH₃-AgNO₃ on gentle warming; *diphenylhydrazone*. Boiled 1 hr. with 4 cc. H₂SO₄ and 15 cc. H₂O, 1.3 g. of the ester gives 0.4 g. *3-methylcyclohexane-1,4-dione*, silvery leaflets from ligroin, m. 50°, sublims undecomd. on heating *in vacuo*, gives no color with FeCl₃; *disemicarbazone*, m. about 240° (violent decompn.). *3-Methylcyclohexan-1-one-4-ol-3-carboxylic acid*, m. 154°, is obtained by shaking 10 g. of the above ester in 25 cc. ice-cold 2 N NaOH 15 min. in a closed bottle with 100 g. of 2.5% Na-Hg, decomp. with 35 cc. of iced 5 N H₂SO₄, extg. 6 times with Et₂O and boiling 1 hr. with 40 cc. of 16% H₂SO₄, the viscous sirup which remains on evap. the Et₂O and boiling gives a strong red color with aq. FeCl₃ and weakly reduces Fehling soln. on boiling.

C. A. R.

Constitution of the peppermint ketone of eucalyptus oils. L. GIVAUDAN and Co. *Perf. Essent. Oil Rec.* 12, 80–1(1921).—Reference is made to the article by Smith and Penfold (cf. C. A. 15, 572) relative to piperitone, which as the authors show possesses the

constitution of a 3-menthenone, leaving it undecided as to the double bond, *i. e.*, whether Δ^1 - (I), or Δ^4 , β -menthenone (II).



While both forms on oxidation would lead to PrCO_2H , the fact that diosphenol also appears as one of the oxidation products would prove its structure to be that of I. W. O. E.

The cyanampholates of the cresols and their reduction products. L. PALFRAY. *Compt. rend.* 172, 980-2(1921).—Cyanocamphor will condense with the three cresols in the same manner as it does with other aliphatic and aromatic alcs. Cyanocamphor and Na cresylate in slight excess are heated in a bomb tube or autoclave at 200–20° for 24 hrs. The condensation product is obtained by taking up in water, extg. with Et_2O , washing with NaOH and distg. *in vacuo*. The *o*-deriv. is a colorless, heavy oil, b_d 242–5°, and crystals from ether-ligroin in beautiful transparent laminas, m. 44–5°, sol. in the majority of org. solvents. The *m*-deriv. is an oily liquid b_d 250°, sepg. after long standing in small crystals m. 104–5°. The *p*-deriv. is the easiest to obtain (70% yield), b_d 248–50° and solidifies immediately, sol. in C_6H_6 but difficultly sol. in Et_2O and abs. alc. On cooling, pure crystals are formed m. 119°, $[\alpha]_D^{25}$ 170° 32' in C_6H_6 . Reduction with Na in abs. alc. gave the best results with the *o*- and *p*-derivs. as the *m*-compd. could not be obtained in cryst. form. A viscous oil, b_d 172° as a reduction product, was obtained in cryst. form after repeated recrystns. from Et_2O or C_6H_6 , m. 76–7°, $[\alpha]_D^{25}$ 68° 43' in C_6H_6 , called α -aminohomocampholic alcohol (A), $\text{C}_6\text{H}_{14}(\text{CH}_2\text{CH}_2\text{NH}_2)\text{CH}_2\text{OH}$. A forms normal HCl, H_2SO_4 and H_2PtCl_6 salts in cryst. form and absorbs CO_2 from the air, giving the carbonate salt, insol. in Et_2O , as a white powder which can be kept only in a dry atm.

R. CHESTER ROBERTS

Chemistry of pinene. I. A new partial synthesis of pinene from a pinene derivative. L. RUZICKA AND H. TREBLER. *Helvetica Chim. Acta* 3, 756–61(1920).—It is possible to prep. pinene from a few of its derivs. which still contain the unchanged 4-membered ring (tetrocane skeleton). Two methods are possible: that of Wallach (*C. A.* 4, 186), who obtained a 10% pinene mixt. by the distn. of nopinolacetic acid, and that of Chugaev (*J. Russ. Phys. Chem. Soc.* 39, 1324(1908)), who decompd. pinocampophyllanthogenate. Another case of the first kind seemed to be the decompn. of pinocampophylltrimethylammonium hydroxide. Pinene resulted in addition to pinocampophyllidimethylamine. The starting material, pinocampophyllamine, Wallach, *Ann.* 313, 367(1900); Tilden, *J. Chem. Soc.* 89, 1560(1906) was prepd. by reducing pinylamine catalytically. Wallach (*Ann.* 253, 251(1889) in prepg. pinene nitrosochloride (A) used a mixt. of pinene, AcOH, EtNO_2 and concd. HCl. R. and T. found that when alc. HCl is used the reaction is smoother. 50 g. pinene and glacial AcOH are cooled, and treated with 60 g. EtNO_2 during 4 hrs., then with 35 cc. 20% alc. HCl. A seps. quickly; it is filtered after it has stood on ice for 12 hrs., and is then washed with alc.; it m. 107–8°. 60 g. nitrosopinene in 400 cc. AcOH and 100 cc. H_2O with 300 g. Zn dust were heated 8 hrs. on the steam bath. The resulting pinocampophyllone, steam distd., extd. with Et_2O and dried with KOH, b_d 90°. Yield 13 g. The residue in the steam-distn. flask, filtered from Zn and treated with NaOH, yields 28 g. pinylamine (B) b_d 90° 25 g. B in 100 g. alc. with 2 g. Pt black reduced catalytically (Tilden used Na and alc.; *J. Chem. Soc.* 89, 1560(1906), gave pinocampophyllamine, b_d 90°, as a colorless oil. The acetate, picrate, and hydrochloride were prepd. and the m. ps. found to be different from those of the compds. prepd. by Wallach and Tilden. Pinocampophylltrimethylammonium iodide (C), prepd. by exhaustive methylation of B, crystals from CHCl_3 and Et_2O in needles, m. 255°, easily sol. in CHCl_3 ,

H₂O, alc., AcOH, but practically insol. in Et₂O and petr. ether. C was transformed into the ammonium base with excess AgOH, the H₂O removed *in vacuo* and the residue heated to 150°. The products were treated with AcOH since one of them was basic and the other neutral. The latter was taken up in Et₂O and when dried with Na gave a pinene, *b*_m 155–6°. The AcOH soln. of the basic product when treated with Et₂O and NaOH yielded a basic material which with MeI gave an *isomeric form* of C, *m.* 300–1°. II. The preparation of homocamphoric acid from pinonic acid; transformation of pinonic acid into tetrahydrocarvone. *Ibid* 762–81.—A compd. of the pinene type has hitherto not been made either from a monocyclic or from a bicyclic compd. Perkin sought in vain to prep. nopinone from $\text{CH}_3\text{CH}_2\text{COCH}_2\text{CH}_2\text{CH}(\text{CMe}_2)\text{Br}$ (*C. A.* 2, 1008) by

splitting off HBr; and Stark (*C. A.* 7, 82) stated that Ca hexahydroisophthalate should give on distn. a ketone but the product described corresponds with a monocyclic compd., 1-methyl-3-cyclohexen-2-one. It is necessary to introduce a new principle for the prepn. of the tetrocean system, *viz.*, a reaction with homopinocamphoric ester analogous to the Na condensation of homocampboric ester (cf. *C. A.* 15, 669). In seeking to prep. homopinocamphoric acid, R. and T. proposed to start from pinonic acid (A). The catalytic reduction of the Et ester of A and treatment of the resulting pinolic ester with PCl₅ gave only a small amt. of the chloro ester, $\text{MeCHClCH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CO}_2\text{Et})\text{CMe}_2$.

This product should give with KCN, followed by hydrolysis, homopinocamphoric acid. But neither in the case of the Cl nor the I ester was it possible to obtain satisfactory yields of the CN compd. *dl*-Pinonic acid was prepd. in general according to Baeyer's method (*Ber.* 29, 22(1896)): 600 g. pinene in 4 l. H₂O were heated 6 hrs. with 1400 g. KMnO₄ in 16 l. H₂O, the excess KMnO₄ was decompd. with MeOH, the MnO₂ filtered off, the alkali neutralized with HCl, the soln. evapd. to 4 l. and then most of the oxidation product extd. by shaking with Et₂O. One crystn. from H₂O was sufficient. The residues of A remaining in the soln. are recovered most satisfactorily by esterifying them with alc. HCl. The distn. of A is accompanied by loss even *in vacuo*. The mixt. of *r*-pinonic ester from French turpentine oil and *d*-pinonic ester from American oil gave *dl*-pinonic ester and sapon. with alc. KOH gave *dl*-A. A with 50% H₂SO₄ gives the ketolactone $\text{AcCH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CO}_2\text{CMe}_2)$ (Baeyer, *Ber.* 29, 326(1896), and like-

wise when treated with 20% HCl. However, it could be esterified as follows: 60 g. A in 200 cc. abs. alc. with 20 cc. HCl-satd. alc. were heated 8 hrs., freed from the alc. by distn., finally *in vacuo*, treated with soda, and extd. with Et₂O and distd., giving 60 g. of the Et ester (F) *b.* 100°. The semicarbazone (D) *m.* 136°. F was prepd. also from A by treatment with Na and EtI. Tiemann and Semmler's procedure (*Ber.* 30, 409 (1897)) for the prepn. of pinolic acid (B) from A with concd. alc. KOH is intricate. R. and T. obtained 50% yields using 16 g. A at 180°. Pinocamphenic acid is formed when A is distd. *in vacuo*. Much of the crude acid remains as a tar in the flask. With a view toward prepg. B, R. and T. used Na-Hg. This acts slowly on A, and is always present in the B produced. 40 g. A were treated with 43 g. Na in 100 cc. alc. and, in the course of 2 hrs., with 450 cc. alc., freed from alc. by distn. after the Na was dissolved, almost neutralized with HCl, concd. on the steam bath, acidified with strong HCl and extd. with Et₂O. The residue could not be crystd. and when distd. *in vacuo* gave tarry products. The noncryst. distillate when oxidized with KMnO₄ gave A. The crude B gave a 60% yield of ethyl pinolate (C), a colorless oil, *b.* 100°. B in alc. was treated with a slight excess of NaOEt and EtI for 8 hrs. in bomb tubes. From the oxidation products of this ester D was prepd. A as the ester (60 g.) was shaken in AcOH with Pt black, treated with H₂O, extd. with Et₂O, shaken with soda and distd. The yields of C and B were nearly quant. 54 g. C were chlorinated in 200 cc. abs. Et₂O with 66 g.

PCl_5 , below 0° , allowed to stand 2 hrs. after the PCl_5 was dissolved, and HCl had been evolved entirely, poured on ice and the Et_2O soln. digested with soda, then dried with Na_2SO_4 . Distn. gave a fraction containing a Cl compd. This interacted with alc. KCN , losing HCl and with alc. NaI , yielding an I ester. These products were not investigated. 125 g. **A** in 300 cc. H_2O and 340 g. KCN in 600 cc. H_2O treated in the course of 2 days with 500 cc. HCl in small portions below 5° , acidified with HCl , extd. with Et_2O , filtered, dried with Na_2SO_4 and coned. *in vacuo*, yielded *pinonic acid cyanohydrin* (**E**), crystals from C_6H_6 , m. 94° , which in cold abs. ether treated with an Et_2O soln. of SOCl_2 , allowed to stand some hrs., heated 6 hrs. on a steam bath, treated with alc., again boiled and finally shaken with ice water and soda soln., gave *ethyl pinonate cyanohydrin*, b.₃₄ $150-2^\circ$. **E** in cold CHCl_3 treated with PCl_5 below 5° , then after standing in the cold, with alc. and ice, extd. with CHCl_3 and treated with soda gave on distn. a fraction b.₃₄ $120-50^\circ$ which when heated with quinoline in xylene for 12 hrs., cooled, and treated with HCl and then soda, gave *ethyl 2-cyano-6-methyl-2,6-heptadiene-5-acetate* (**G**), a yellow oil, b. $120-40^\circ$. When this was treated with 50% KOH and alc. and the mixt. was heated for 4 days on the steam bath, a product resulted which after acidification and extrn. with Et_2O formed a yellow cryst. mass of a di- CO_2H acid; the Na salt when treated with EtI at 120° in bomb tubes and extd. with Et_2O gave *diethyl 2-carboxyl-6-methyl-2,6-heptadiene-5-acetate* (**I**), a colorless oil b.₃₄ 115° . The di-Me ester, prepd. with Me_2SO_4 is a colorless oil, b.₃₄ 115° . **H** was heated in bomb tubes for 12 hrs. at 180° with Ac_2O and the product was distd. *in vacuo*. The product, considered to be carvacryl acetate, when sapond. with KOH for 15 hrs. gave carvacrol. **I** was reduced in MeOH with Pt to *diethyl methylisopropylpimelate*, a colorless oil, b.₃₄ 115° . The dimethyl ester (**J**) b. 108° . No solid products could be obtained when the esters were sapond. **J** was treated in xylene with Na for 5 hrs., then HCl was added; the xylene was removed and the H_2O layer was treated with Et_2O . The Et_2O ext. and the xylene were combined, dried with Na_2SO_4 , and the solvents were removed *in vacuo*. The product heated 15 hrs. with 10% KOH gave *dl-tetrahydrocarvone* (Wallach). The semicarbazone and oxime were prepd.

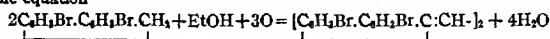
H. E. WILLIAMS

Phenheptamethylene and some other compounds of the phenheptamethylene group. W. BORSCHKE AND A. ROTH. Univ. Göttingen. *Ber.* **54B**, 174-7 (1921).— $\text{PhCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{COCI}$ (10 g.) in 100 cc. CS_2 slowly treated in the cold with 6.7 g. AlCl_3 and allowed to stand overnight (protected with a CaCl_2 tube) gave about 4 g. α -ketophenheptamethylene (Kipping and Hunter, *J. Chem. Soc.* **79**, 602 (1901)), 3 g. of which, boiled 12 hrs. with 150 cc. of 25% HCl and 32 g. amalgamated Zn (75 cc. fuming HCl being added during the boiling) yielded 1.1 g. *phenheptamethylene (benzosuberene)*, mobile oil of petroleum-like odor, b.₃₄ 217° , stable to KMnO_4 . Similarly *methylenedioxybenzosuberene*, b.₃₄ $150-5^\circ$, m. $69-70^\circ$, is obtained by reduction of the ketone (**B**) (C. A. **8**, 2710). *Benzalbenzosuberone*, from 2.8 g. **A** and 1.6 g. BzH in 10 cc. alc. treated with a few drops of NaOEt , needles from $\text{EtOH-H}_2\text{O}$, m. 82° . *Isonitrosobenzosuberone*, yellowish needles from CCl_4 , m. $133-4^\circ$, is obtained by treating 1.6 g. **A** in 10 cc. dry Et_2O with 0.23 g. Na shavings, then in the cold with 1.17 g. iso- AmONO , letting stand in the ice chest until all the Na is dissolved, adding an equal vol. of ice H_2O , removing the Et_2O layer, extg. once more with Et_2O , adding an excess of AcOH to the aq. soln. and taking up the pptd. oil in Et_2O . *Benzalmethylenedioxybenzosuberone*, from **B**, plates from MeOH , m. $120-1^\circ$. *Anisal analog*, needles from CHCl_3 , m. $159-60^\circ$. *Piperonylidene compound*, yellowish crystals from $\text{CHCl}_3\text{-EtOH}$, m. $184-5^\circ$. The oxonium chloride of *o*-hydroxybenzalmethylenedioxybenzosuberone is obtained as the *ferric chloride double compound*, $\text{C}_{15}\text{H}_{10}\text{O}_4\text{Cl}_2\cdot\text{FeCl}_3$, dark red leaflets from AcOH-HCl , m. 159° , from 2.04 g. **B** and 1.22 g. *o*- $\text{HOC}_6\text{H}_4\text{CHO}$ in 10 cc. cold AcOH treated 0.5 hr. with HCl gas and, after some days, with solid FeCl_3 .

C. A. ROUILLER

Fluorene. K. STAHRFOSS. *Bull. soc. chim.* [4] **29**, 142 52 (1921).—The work was

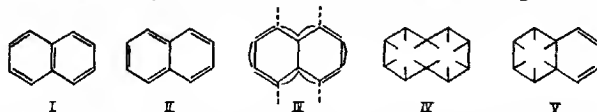
undertaken to det. the effect of introducing Br atoms or NO₂ groups into fluorene on the ease of formation of the alkali metal salts. Refluxing 2,7-dibromofluorene (A) with alc. NaOH or KOH converts it into *sym*.-tetrabromodiphenylenebutadiene (B) according to the equation



A also condenses with 2,4-(O₂N)₂C₆H₃Cl and *p*-H₂NC₆H₄OH in alk. soln., giving red products. S. speculates on the structure of these compds. but gives no exptl. data to establish his formulas. 10 g. A suspended in 100 g. EtOH and refluxed for a few hrs. on a water bath with 0.5 mol. aq. NaOH (or KOH) gives B as a red ppt., which can be crystd. from PhNH₂. B does not melt nor sublime at 450°. B is very stable toward reagents such as concd. HNO₃ and concd. H₂SO₄. When treated with a mixt. of these two acids B gives a colored soln. which S. thinks may contain a compd. with four NO₂ groups added on to the double bonds in B.

CARL S. MARVEL

Spectrochemistry of polynuclear aromatic compounds and the constitution of naphthalene. KARL V. AUWERS AND ADELHEID FRÜHLING. *Ann.* 422, 192-230 (1921).—C₁₀H₈ and the following derivs. were examd.: α-Me, β-Me, 1,2-di-Me, 1,6-di-Me, α-Cl, β-Cl, α-Br, 1,2-di-Cl, 1,4-di-Cl, α-C₁₀H₇OEt, β-C₁₀H₇OEt, 2,3-di-Am ether, 2,7-di-Am ether, α-C₁₀H₇CO₂Et, β-C₁₀H₇CO₂Et, di-Et naphthalate, 6,1-C₁₀H₆MeCO₂Me, 1,2-C₁₀H₆(OMe)CO₂Et, 3,2-C₁₀H₆(OMe)CO₂Me, 4,1-C₁₀H₆(OMe)CHO, acenaphthene, 2,3-Dihydroxynaphthalene diamyl ether, pale yellow oil, b₇₀ 229-30°. 2,7-Dihydroxynaphthalene diamyl ether, fine, long needles from benzene, m. 75°, b₁₈ 236-7°. Ethyl 1-methoxy-2-naphthoate, b₁₄ 184-5°. 3-Methoxy-2-naphthoic acid, small yellowish crystals, m. 133-4°. Ethyl ester, m. 18° (approx.), h₁₈ 208.5°. The results are given in a series of tables, for which the original should be consulted. They show a remarkable uniformity in the sp. exaltations and a moderate increase in the dispersion. The values show again that each typical class of compds. has a typical spectrochem. behavior. In general, substituents are optically of little influence in the C₁₀H₈ series, hut the halogens furnish an exception to this rule, the exaltations for the halogenated C₁₀H₇ being only about 0.5 of that for the parent substance, although the dispersive power remains virtually unaltered. With the exception of 4-MeOC₁₀H₆CHO, no deriv. shows a higher exaltation than C₁₀H₈ itself, so that the action of substituents in the C₁₀H₈ mol., unlike that in the C₆H₆ mol., is mostly depressive. The influence of position is slight, although the optical exaltations are, on the average, somewhat higher with the β- than with the α-derivs. In its optical const. acenaphthene corresponds perfectly with mono- and dialkynaphthalenes, the rule that the formation of a satd. ring does not alter spectrochem. character being thus obeyed. The bearing of these results on the various structures which have been proposed for the C₁₀H₈ mol. is discussed at length. The con-



clusion is reached that formula I, as originally interpreted by Erlenmeyer, and also formulas III, IV and V are incompatible with the spectrochem. data. No choice is, however, yet possible between formula I, if this be taken to represent C₆H₄ with an unsatd. cyclic side, chain, and formula II, expressing a hydroaromatic structure. The following compds. containing 2 C₆H₅ nuclei, variously united, have been examd.: Ph₂CH₂, MeC₆H₄CH₂Ph, MeC₆H₄CH:CHPh, Ph₂, (o-EtOC₆H₄)₂, diphenylene oxide, 9-methylfluorene, and fluorenone. Ph₂CH₂ behaves spectrochem. as a hydrocarbon of the C₆H₅ series, hut when the chain between the 2 rings is unsatd., as in MeC₆H₄CH:CHPh, marked exaltation appears; the latter is also the case with Ph₂ and its EtO

deriv. Since the exaltation in the tricyclic compds. is higher than that of Ph₃ fluorenone exhibiting unexpectedly high exaltation, doubt is cast on the presence of 2 true C₆H₅ nuclei in these mols. The double linkings of the 2 external rings no longer exist in a condition of optical neutralization, but have assumed a more or less olefinic character. The same change is seen in a comparison of the chem. properties of Ph₃CH₂ and fluorene; in the former the CH₂= is chem. indifferent, while in fluorene the H atoms of the uniting CH₂= group are highly reactive. *m*-Methylstilbene, PhCH:CHC₆H₄Me, prepd. by the slow distn. of C₆H₅CH:CHCO₂C₆H₄Me, stout crystals, m. 52.5-3.5°, b₂₀ 206-7°.

C. J. Wessr

Azodicarboxylic ester as a means for introducing the hydrazine residue into the aromatic nucleus. I. Azo ester and β -naphthylamine. OTTO DIEELS. Univ. Kiel. Ber. 54B, 213-26(1921).—From all earlier work it would seem that in general primary and secondary amines convert (EtO₂CN)₂ (A) into substituted azodicarboxamides while tertiary amines form peculiar addition products of the type PhNMeCH₂N(CO₂Et)NHCO₂Et (C. A. 7, 2949). This, however, is not true, for primary aromatic amines also add, in part quant., to A. It appears in every case that the addition takes place in two phases which proceed with different velocities, depending on the nature of the amine; first there are always formed deep brown-red or dark red solns. from which then the well crystd. colorless addition products sep. Probably there are first formed colored salts of A, possibly with a quinquevalent N atom, which rearrange with different velocities into the more stable colorless products. Moreover, these show varying degrees of stability towards chem. reagents. The addition products of PhNH₂ and the toluidines have no tendency to undergo further transformations and it is possible to convert these substances, in themselves stable, only into their components or their transformation products, so that in these cases the union between the two components is apparently very loose. The addition product of β -C₁₀H₇NH₂, on the other hand, is formed exceedingly smoothly and quickly and no decompn. into its components has ever been observed in its numerous reactions. From all the observations which have been made the free NH₂ group seems to be involved in the addition reaction. Possibly in many cases a triazan deriv. is formed: RNH₂ + A = RNHN(CO₂Et)NHCO₂Et, but with β -C₁₀H₇NH₂ there is no doubt that the product contains an intact NH₂ group, as shown by acetylation, formylation, salt formation with acids and diazotization. It must be assumed, therefore, that the union is effected through a H atom of the C₁₀H₇ nucleus, possibly that in position 1, the addition product having the structure 2,1-C₁₀H₆(NH₂)-N(CO₂Et)NHCO₂Et (B). The presence of a free NH₂ group is shown beyond doubt by the behavior of B towards COCl₂ (see below) and that the addendum is in position 1 and not 3 as shown by the action of HI in AcOH and of H₂O₂ in AcOH. Thus, the structure of B is established with certainty but further study will be required to clear up the mechanism of the reaction whereby it is formed and the reason for the difference in behavior of the PhNH₂ and toluidine addition products on the one hand and B on the other. *2-Amino-1-dicarboxyethylhydrazinonaphthalene* (B) (4 g. from 2 g. β -C₁₀H₇NH₂ in 5 cc. warm alc. treated rapidly with 2.5 g. A in several portions), leaflets from alc., MeCN, 50% AcOH or other solvents, m. 198°, forms difficultly sol., well crystd. salts easily hydrolyzed by H₂O; *hydrochloride* (2.3 g. from 2 g. B and 3 cc. of 37% HCl), light violet crystals, smoothly regenerates B on boiling with H₂O; *2-acetylaminonaphthalene*, from 2 g. B boiled up with 7 cc. Ac₂O, needles from abs. alc., m. 218-9°. *Addition product of α -C₁₀H₇NH₂ and A*, m. 168°; *acetyl derivative*, m. 132°. When 4 g. B is added to 28 g. KOH in 10 cc. boiling H₂O, EtOH is at once split off; after 2-3 mins. 80 cc. hot H₂O is added and on acidification with AcOH there is deposited 3.5 g.

of a compound C₁₀H₆ $\begin{matrix} \diagup N(CO_2Et) \\ \diagdown NH-CO \end{matrix}$ NH (C), silvery prisms from alc., m. 265° (violent

decompn.), sol. in cold dil. NaOH with bluish fluorescence, reprecipitated unchanged by acids and insol. in excess of acids; it is also obtained in 3-g. yield from 5 g. B boiled 6 hrs. under a reflux with 15 cc. piperidine (which has not been dried with special care) and then acidified with 50% AcOH. If 5.5 g. B is boiled 2 hrs. under a reflux with 27.5 cc. of 30% KOH and 55 cc. of 95% alc., cooled, diluted with H₂O and acidified with AcOH there seps. 3 g. of a compound $C_{10}H_8NH.NH.CO.NH$, light brown prisms with bronze

luster from AcOH, m. 315–20°, also formed when C is boiled 2 hrs. under a reflux with concd. alc. KOH. B (10 g.) heated in a CO₂ atm. with 20 cc. AcOH until dissolved, then treated with 25 cc. colorless HI (d. 1.96) and very slowly distilled in a current of CO₂ for 40–5 min., the temp. finally rising to 93°, gives 3 g. EtI (in the distillate), while the residue, after standing several hrs. in ice, yields 2.5 g. of 2-methyl- β -naphthimidazole-HI, silvery crystals from 50% AcOH, converted by aq. NH₄OH into the free base, thick hard crystals from C₆H₆, m. 169°. Two g. B in 5 cc. of 37% HCl at 0° with 0.45 g. NaNO₂ in 35 cc. H₂O yields a brown-yellow soln. of the diazonium salt, which on gentle warming evolves N and deposits an amorphous brown-yellow mass and with alk. β -naphthol or Na naphthionate couples to form azo dyes, the last of deep dark Bordeaux color and sol. in hot alc. with fuchsin-red color. From 1 g. B in 30 cc. C₆H₆ treated almost boiling with 10 cc. of a soln. of COCl₂ in PhMe is obtained 1 g. of a *carbamyl chloride*, $C_{10}H_8(NHCOCl)N(CO_2Et)NHCO_2Et$, leaves, m. about 145°, dissolves clear in MeOH but the soln. quickly becomes turbid and deposits the *methyl ester*, scales from AcOH, m. 207–8°; *ethyl ester*, m. 154°. The chloride (3 g.) heated about 1 hr. in a xylene-vapor bath, loses HCl and leaves 2.7 g. of a transparent, light brown, colophony-like, brittle mass which, boiled 2 min. with AcOH, dissolves clear and on cooling there seps. a yellowish white, granular *product*, $C_{11}H_9O_2N_2.AcOH$, m. 127–8°, converted by dissolving in boiling MeOH or EtOH and cooling into a *compound*, $C_{10}H_8N(CO_2Et)N(CO_2Et).CO.NH$, m. 180–1°, regenerates the AcOH compd. when

heated with AcOH and 3 g. heated 6 hrs. under a reflux with 6 cc. moist piperidine and acidified with AcOH yields 1.5 g. C. From 101-g. portions of B gently boiled 5–6 min. each with 2 cc. AcOH and 1.25 cc. of 30% H₂O₂ is obtained 8–9 g. of brownish crystals of a compound $C_{10}H_8N(CO_2Et)N(CO_2Et).NOH$, purified by cautiously and quickly dis-

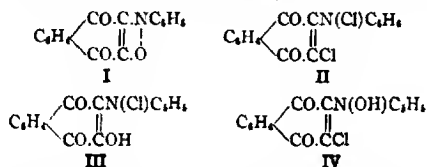
solving in small portions in warm MeOH (whereby a large part is destroyed); the 1 g. brown-yellow crystals sepg. on cooling are cautiously recrystd. from a little warm MeCN from which they sep. in stout brown-yellow prisms with blue luster, m. 96° (decompn.), 0.5 g. of which gently warmed with 1.25 cc. of certain samples of MeCN (probably containing traces of impurities) evolves CO₂, dissolves and, on cooling, yields *carboxyethylazimidonaphthalene*, $C_{10}H_8N(CO_2Et).N:N$, thick, faintly brownish prisms,

m. 124–5°, identical with the product obtained in 2.1 g. yield from 2.25 g. 1,2-naphthyleneazimide in 22.5 cc. boiling C₆H₆ and 1.05 g. C₆H₅N slowly treated with 1.45 g. ClCO₂Et and boiled 5 min. longer.

CHAS. A. ROULLER

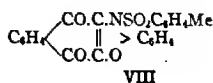
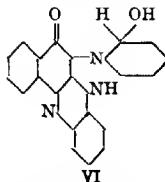
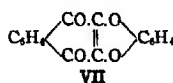
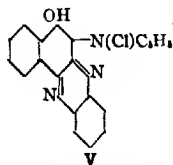
2,3-Dichloro- α -naphthoquinone. FRITZ ULLMANN AND MARGARETE ETTISCH. Univ. Berlin. Ber. 54B, 259–72(1921).—2,3-Dichloro- α -naphthoquinone (A), long yellow needles from boiling AcOH containing a little CrO₃, m. 193°, is obtained in 37.2 g. yield from 50 g. finely powdered α -naphthol heated 2 hrs. at 50° with 90 cc. concd. H₂SO₄, poured into 300–400 cc. ice H₂O, dild. to 500 cc., treated with 250 cc. crude concd. HCl, then with about 75 g. finely powdered KClO₃ in 0.5-g. portions (the temp. being kept at 70° when it reaches this point), kept 1 hr. longer at 70°, cooled, filtered, washed to neutrality and treated in boiling AcOH with a little Cl. When 6 g. A are gently warmed with 9.4 g. C₆H₅N on the H₂O bath until the brown-yellow soln. has become green and has started to deposit a brown resinous mass, then removed from the bath until the

reaction is over, dild. with 20 cc. alc. and again heated to boiling the resinous mass changes into a yellow cryst. substance (5.73 g.), m. 292°, sepg. from H₂O in bitter, light yellow felted needles, sol. in concd. H₂SO₄ with red-brown, in concd. HCl easily with light yellow color and repptd. by much H₂O, insol. in cold dil. NaOH, sol. on boiling and sepg. unchanged on cooling, deliquesces in glacial AcOH, gives a light brown vat with NaOH and Na₂S₂O₄, mol. wt. in freezing PhOH 253.9. The compd. is *3-hydroxy- α -naphthoquinone-2-pyridinium anhydride* (I). In its prepn. the A and C₆H₅N probably first add to form a compd. II, which under the influence of H₂O or alc. is acted upon by the excess of C₆H₅N in its capacity as a base with the formation of III or IV, which by loss of HCl gives I. From 0.85 g. I in 100 cc. boiling H₂O treated dropwise with a concd. soln. of 1 g. KMnO₄ are obtained 86% of C₆H₅N and 71% phthalic acid.



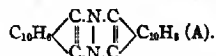
Phenyldrazone (1.3 g. from 1 g. I and 1 g. PhNHNH₂ in 20 cc. AcOH heated 1 hr. on the H₂O bath), yellow-red needles from alc., m. 253°, insol. in NaOH, sol. in concd. H₂SO₄ with blood-red, easily in C₆H₆ with yellow color, deliquesces in AcOH and PhNO₂. *Oxime* (0.76 g. from 1 g. I and 0.4 g. NH₂OH.HCl in 100 cc. H₂O on the H₂O bath), yellow felted needles, m. about 244°, sol. in hot HCl and alc. with yellow, in concd. H₂SO₄ with brown-red, in dil. NaOH with blood-red color, deliquesces in C₆H₆, AcOH and PhNO₂. From 0.5 g. I heated 2 hrs. on the H₂O bath with 0.5 g. *o*-C₆H₄(NH₂)₂ and 20 cc. of 10% HCl is obtained 0.59 g. *1-hydroxynaphthazine-2-pyridinium chloride* (V), red needles with metallic luster, m. about 305°, only slightly sol. in boiling H₂O with yellow color; on addition of a little HCl to the aq. suspension it dissolves easily with brown-yellow color and formation of a dibasic salt which is again hydrolyzed by diln. with H₂O; boiled with aq. KMnO₄ it evolves the odor of C₆H₅N; it dissolves easily in AcOH with yellow-brown, in PhNO₂ with orange-red, in concd. H₂SO₄ with blood-red color; with NaOH it loses its Cl as NaCl, the red crystals becoming yellow. Probably the true ammonium base is first formed, but the properties of the product, especially its soly. in PhNO₂, indicate that it rearranges into the stable isomer VI, yellow felted thread-like crystals from alc., m. about 310°, sol. in cold PhNO₂ with orange-yellow color changing to orange-red on warming, reconverted into V by HCl. *3-p-Toluenesulfonylamino- α -naphthoquinone-2-pyridinium anhydride* (1.4 g. from 1 g. A, 1 g. *p*-MeC₆H₄SO₂NH₂ and 5 cc. C₆H₅N boiled 5 min. and heated 0.5 hr. longer on the H₂O bath, or from 1 g. A cautiously warmed with 5 cc. C₆H₅N until the green intermediate product begins to sep. and then slowly heated to boiling with 1 g. MeC₆H₄SO₂NH₂, m. 250°, sol. with blood-red color in PhNO₂, from which it seps. in light red needles, converted by dil. HCl into a yellow salt hydrolyzed on diln. with H₂O, sol. in concd. H₂SO₄ with light brown color, gives with NaOH-Na₂S₂O₄ a brown vat which, however, has no affinity for cotton; 0.2 g. heated on the H₂O bath 1 hr. with 3 cc. concd. HCl evolves the odor of *p*-MeC₆H₄SO₂Cl and yields 0.09 g. I, while 0.5 g. boiled with 5 cc. of 33% NaOH and 20 cc. H₂O gives 0.25 g. I and 0.15 g. *p*-MeC₆H₄SO₂NH₂. Attempts to condense A with MeC₆H₄SO₂NH₂ in AmOH by means of K₂CO₃ gave only 2-chloro-3-hydroxy- α -naphthoquinone. When 1 g. K₂CO₃ in 8 g. boiling PhOH is heated until all the H₂O is driven off, then cooled, cautiously warmed 1 hr. on the H₂O bath with 1 g. A, dild. with alc., boiled up and filtered hot, there is obtained 1.45 g. *2,3-diphenoxy- α -naphthoquinone*, orange-yellow felted needles from xylene, m. 205°, sol. in concd. H₂SO₄ with brown color.

forms a yellow $\text{Na}_2\text{S}_2\text{O}_4$ vat, which has no affinity for cotton. α -Naphthoquinone-2,3-benzodioxine (VII) (0.63 g. from 1 g. o - $\text{C}_6\text{H}_4(\text{OH})_2$, 5 cc. NH_4Cl , and 1 g. A heated 4 hrs. on the H_2O bath), long red-violet needles from xylene, m. 280° , easily sol. in boiling $\text{C}_6\text{H}_5\text{N}$, AcOH and PhNO_2 with red, in concd. H_2SO_4 with dark green color, repptd. by H_2O in light red flocks. α -Naphthoquinone-1,2-anthraquinonedioxine (0.6 g. from 0.72 g. alizarin, 4 cc. $\text{C}_6\text{H}_5\text{N}$ and 0.44 g. A heated 30 min. on the H_2O bath), m. about 375° , sol. in H_2SO_4 with blood-red color changing to brown on heating, gives with NaOH - $\text{Na}_2\text{S}_2\text{O}_4$ a brown-red vat dyeing cotton in red-brown shades. *N*,*p*-Toluenesulfonyl- α -naphthoquinonephenoxazine (VIII) (1.6 g. from 1.3 g. o -(*p*- $\text{MeC}_6\text{H}_4\text{SO}_2\text{NH})\text{C}_6\text{H}_4\text{OH}$ in 2.5 cc. $\text{C}_6\text{H}_5\text{N}$ and 1.15 g. A after 0.5 hr. on the H_2O bath), leaflets with golden luster from alc., decomps. about 130° into a blue substance which m. about 320° , gives with NaOH - $\text{Na}_2\text{S}_2\text{O}_4$ a yellow vat having but little affinity for cotton; 0.6 g. in 5 cc. cold concd. H_2SO_4 treated dropwise with about 50 cc. H_2O yields 0.35 g. α -naphthoquinonephenoxazine, deep blue crystals from xylene or PhCl . 2-Amino-3-chloro- α -naphthoquinone (1.05 g. from 1.5 g. A in 10 cc. boiling PhNO_2 satd. with dry NH_3), yellow-brown needles from AcOH , m. 193° , easily sol. in concd. HCl , converted by boiling dil. NaOH , with loss of NH_3 , into the 2-chloro-3-hydroxy compd. 1,4,2- $\text{C}_{10}\text{H}_5(\text{OH})_2\text{SC}_6\text{H}_4\text{CO}_2\text{H}$ (Ghosb and Smiles, C. A. 8, 2682), m. (crude) 208° , is obtained in 2.3-g. yield from 1.6 g. o - $\text{HSC}_6\text{H}_4\text{CO}_2\text{H}$ in 10 cc. warm Me_2CO treated with 1.5 g. α -naphthoquinone in small portions, filtered from the 0.4 g. $(\text{HO}_2\text{CC}_6\text{H}_5)_2$ which seps. on boiling, treated with a little aq. H_2SO_4 , heated to boiling and dild. with H_2O ; 1.5 g. boiled 5 min. in 5 cc. Ac_2O with 0.7 g. ZnCl_2 , cooled somewhat and cautiously treated with about 1 cc. H_2O gives 1.75 g. 1,4-diacetoxynaphthothioxanthone, long red needles from AcOH , m. 208° . G. and S. claim to have obtained this compd. in yellow needles, m. 229° , by treating their acid with Ac_2O ; by G. and S.'s method U. and E. obtained only colorless crystals, m. 190° . The diacetate dissolves in concd. H_2SO_4 with blue color becoming olive-green on warming, with evolution of SO_2 ; dild. with H_2O ppts. brown flocks. If the acetate is warmed under NaOH a part dissolves with blue color; on shaking the soln. with air it is quickly decolorized and deposits brown flocks; if air is excluded by adding a few drops of C_6H_6 or PhCl to the mixt. of the acetate and NaOH , which is then warmed without shaking, a deep corn flower-blue soln. results. When 0.23 g. of the acetate is heated under a reflux with 5 cc. alc. and 2 cc. of 10% NaOMe it dissolves with yellow-green color gradually changing to brown and then to blue-green; on shaking the soln. becomes brown and solidifies to a magma yielding 0.14 g. of α -naphthoquinonethioxanthone, long dark brown needles from PhNO_2 , m. about 310° , insol. in NaOH , sol. in H_2SO_4 with green color, converted by boiling with AcOH and SnCl_4 into 1,4-dihydroxy-naphthothioxanthone, Bordeaux-red needles which, gently warmed under Et_2O with dil. NaOH , dissolve with a blue color destroyed by shaking with air. yellow-brown flocks being deposited.



CHAS. A. ROULLER

o-Quinones and 1,2-diketones. I. Elucidation of the anhydride of acenaphthenequinone imide as diacenaphthylene azotide (acenaphthylene-1,2-azine). A. SCHÖNBERG AND F. NEDZATI. Techn. Hochschule Berlin. *Ber.* 54B, 238-42(1921).—Graebe's "acenaphthenequinone imide anhydride, $C_{16}H_{12}ON_2$ " (*Ann.* 276, 9(1893)), has been found to be really *diacenaphthylene azotide (acenaphthylene-1,2-azine)*,

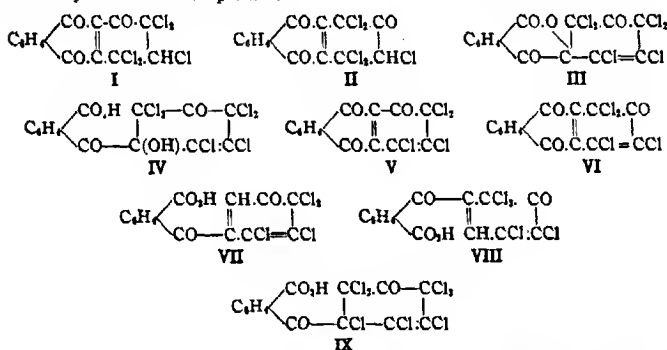


To obtain it in cryst. form, 7 g. acenaphthenequinone are heated 3 hrs. at 100° in sealed tubes with 15-20 cc. concd. NH_4OH and the resulting red mass is drained, well washed, dried at 105° , powdered and crystd. from $PhNO_2$ (50 cc. for each 3 g.) until the product consists of homogeneous red needles free from admixed colorless crystals. A is very difficultly sol. in the ordinary solvents, sublimes undecomposed. (even over Fe filings) at 410° , m. (on the Thiel block) above 400° (decompn.), dissolves in concd. H_2SO_4 with red-brown color, mol. wt. in freezing $PhOH$ 325-37. The sublimation over Fe filings may be used to purify A. A is also obtained, with varying yields, from 8 g. acenaphthenequinone dioxime in 500 cc. alc. treated in the course of 1 hr. with 50 g. $SnCl_2$ in 150 cc. concd. HCl , then made alk. with NH_4OH and allowed to stand several days. II. Action of aqueous ammonia on benzils in the absence of air. A. SCHÖNBERG. *Ibid.* 242-4.—The elucidation of the structure of the supposed acenaphthenequinone imide anhydride (preceding abstr.) led to the investigation of the action of aq. NH_4OH on other diketones whose structure is such that a rearrangement of the keto into an enol group is not possible. In working with the benzils, it was found that by excluding air and alc., triphenyloxazole (A) and its derivs. can be obtained easily and almost quant. Thus, 5 g. benzil heated 7 hrs. at 120° with 10 g. concd. NH_4OH in a sealed tube gives more than 90% A, m. $114-5^\circ$; *p*-anisil gives more than 90% *tri-p-anisilyloxazole*, rodlets from alc., m. 141° ; *tri-p-tolyloxazole* (more than 90% from *p*-tolil), prisms from alc., m. 145° .

CHAS. A. ROUITLER

Degradation reactions in the anthraquinone series. K. FRIES AND W. HARTMANN. Tech. Hochschule Braunschweig. *Ber.* 54B, 193-200(1921); cf. *C. A.* 14, 2199.—It was shown in the earlier paper that the compd. I, obtained by chlorination of $\alpha-C_6H_4(CO)_2C_6H_5NH_2$, is transformed into *o*-[2,3,4,6,5- $Cl_4(HO)C_6CO$] $C_6H_4CO_2H$ (A) when heated with concd. H_2SO_4 . The compd. II, prep'd. by chlorination of $\beta-C_6H_4(CO)_2C_6H_5NH_2$, is unchanged by H_2SO_4 under the same conditions, but in attempts to split off HCl from I and II the conditions have been found under which II also undergoes a transformation involving the rupturing of the central ring. *1,1,3,4,4-Pentachloro-2-keto-1,2,3,4-tetrahydroanthraquinone* (II), from $\beta-C_6H_4(CO)_2C_6H_5NH_2$ in 12 parts $AcOH$ and 1 part fuming HCl treated at room temp. with Cl until no more is absorbed (yield, 93%), yellow leaflets from $AcOH$, m. 213° , gives reduction products (di- and trichloro- β -hydroxyanthraquinone) on long boiling in $AcOH$ or heating in concd. H_2SO_4 , crystals unchanged from concd. HNO_3 , only slowly attacked by alkalis at room temp., quickly on warming, turning brown and undergoing deep-seated decompn., liberates I from KI , reduced in hot $AcOH$ by the calcd. amt. of $SnCl_2$ and HCl to *1,3-dichloro-2-hydroxyanthraquinone*, yellow needles from $AcOH$, m. 208° (yield, 95%), which forms difficultly sol. red salts with $NaOH$, soda and NH_4OH and yields an *acetate*, yellow needles from $AcOH$, m. 179° . *1,3,4-Trichloro-2-hydroxyanthraquinone* (B), from 40 g. II boiled 15 min. in $AcOH$ with 27 g. $NaOAc$, yellow needles from $AcOH$, m. 252° , forms difficultly sol. red salts with alkalis; *acetate*, yellow leaflets or needles from $AcOH$, m. 174° . When 10 g. I or II in $AcOH$ and 20 g. crystd. $NaOAc$ are chlorinated until the soln. does not darken on boiling there is formed the compound III, rhombohedrons from $AcOH$, m. $151-2^\circ$, stable towards soda at room temp., sol. in $NaOH$ with red-brown color and formation of IV (below), gives only traces of $AgCl$ after boiling 0.5 hr. in

AcOH with AgOAc, reduced in AcOH by SnCl₂ on gentle warming to A, which, in turn, regenerates III when chlorinated in AcOH. III in 70 parts alc. at 25° quickly treated with 2 N NaOH until a portion of the soln. remains clear on addition of H₂O, then, after 30 sec., with excess of concd. HCl yields after some hrs. a mixt. of unchanged III and of the acid IV, which is extd. with boiling C₆H₆ and seps. from dil. alc. in needles or stout prisms, m. 204° (decompn.), sol. in soda with violet color changing to red on long standing, in NaOH with orange-red color, does not liberate I from KI in AcOH, reconverted into III by warming in AcOH on the H₂O bath with a few drops of concd. H₂SO₄, decompd. by cautious warming on the H₂O bath in AcOH with excess of SnCl₂ into tetrachlororesorcinol and phthalic acid. The conversion of I into A can be effected not only by heating with H₂SO₄ but also by boiling it in AcOH 1 min. with 3 parts NaOAc. On splitting off HCl, I and II first form the compds. V and VI, but while NaOAc alone ruptures V with formation of VII and reduces VI, forming B, the presence of Cl prevents the reduction and VI yields VIII; in the case of V the rupture of the middle ring occurs at the *o*-, in that of VI at the *p*-position to the CO group of the side ring. VII and VIII both rearrange at once into the same product, A. In the formation of III IX is undoubtedly an intermediate product.



CHAS. A. ROUILLER

Thiophene compounds. I. The isomeric propyl- and isopropylthiophenes. HELMUT SCHREIBLER AND MARTIN SCHMIDT. *Techn. Hochschule Berlin. Ber.* 54B, 139-54 (1921).—It has recently been found that many bituminous tar oils consist chiefly of thiophene homologs and in some of the schist tar oils examd. the propylthiophene fraction seems especially abundant. S. and S. have accordingly undertaken a study of some monopropylthiophenes. *Ethyl γ -bromo- α,α -dimethylacetate* (125 g. from 100 g. AcCM₂CO₂Et with 102 g. Br in the cold), b₃ 114-6°; 94 g. added to 9.1 g. Na wire and 63.5 g. CH₂(CO₂Et)₂, which have been allowed to stand in 300-400 cc. Et₂O until all the Na is dissolved gives, after standing overnight, 86.5 g. (EtO₂C)CHCH₂COCMe₂CO₂Et, greenish yellow thick oil, b₁ 188-9° (Conrad. *Ber.* 30, 864(1897)); 70 g. of this are boiled 9 hrs. with 325 g. H₂SO₄ and 600 g. H₂O, neutralized with NaOH, concd. as far as possible on the H₂O bath, acidified to Congo with dil. H₂SO₄, allowed to stand overnight, filtered, extd. with Et₂O, dried with Na₂SO₄ and evapd.; the resulting thick yellow oil (32 g.) is boiled 3 hrs. under a reflux with 70 g. NaOH in 350 g. H₂O, acidified to Congo with dil. H₂SO₄, extd. with Et₂O, dried with Na₂SO₄ and evapd., giving 26.2 g. Me₂CHCOCH₂CO₂H, m. 41°. From 34.5 g. of the Na salt (prepd. by neutralizing 30.5 g. of the acid with NaOH, evapg. to dryness and drying at 110°) and 80 g. P₂S₅ heated in a retort with a free flame until reaction sets in is obtained 20.3 g. of

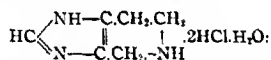
an oily distillate which, taken up in Et_2O , washed several times with NaOH and H_2O , dried overnight with KOH , evapd., boiled over Na under a reflux and fractionated, gives 13 g. of 2-isopropylthiophene (A), b. $152-3^\circ$, d_{20}^{20} 0.9691, d_4^{20} 0.9674, n_D^{20} 1.5030, M_D^{20} 38.53, Σ_D^{20} 30.56, $n_F - n_C$ 0.01447, has a faint benzine-like odor, gives with phenanthrenequinone in AcOH and H_2SO_4 (Laubenheimer reaction) an intense cherry-red ring, and with isatin and concd. H_2SO_4 on warming an intense red-brown color. From 2.5 g. A in 10 g. CS_2 , and 2 g. AcCl added dropwise to 3 g. AlCl_3 in 15 g. cold CS_2 , allowed to stand with frequent shaking 1.5 hrs. in ice and 0.5 hr. at room temp., decompd. with ice and dil. H_2SO_4 , treated with Et_2O , sepd. from the aq. H_2SO_4 layer, washed repeatedly with dil. H_2SO_4 , H_2O , Na_2CO_3 and H_2O again and dried with CaCl_2 , is obtained 2.5 g. 2-isopropyl-5-acetothienone, b. 130.5° , n_D^{20} 1.5428; oxime, crystals from petr. ether, m. 74° ; *p*-nitrophenylhydrazones, dark red needles, m. 198° , gives in alc. with a trace of NaOH an intense dark violet color which again disappears on addition of an acid. 2-Isopropyl-5-benzoylthiophene (2.6 g. from 3 g. A in 15 g. CS_2 and 3.5 g. BzCl added in the course of 15 min. to 3.4 g. AlCl_3 and 15 g. CS_2 in ice, allowed to stand 3 hrs. at room temp. and boiled 15 min. under a reflux, the residue from the Et_2O ext. being boiled 0.5 hr. with concd. Na_2CO_3 to destroy unchanged BzCl), b. $209-10^\circ$. 2-Propionylthiophene (β -propiethienone), obtained in 19-g. yield from 14.4 g. thiophene and 16 g. EtCOCl in 35 g. CS_2 and 25 g. AlCl_3 in 60 g. CS_2 (Meyer, *Thiophen-Gruppe* 1888, 164), h. $225-9^\circ$. 2-Propylthiophene (9.5 g. from 20 g. of the above ketone and amalgamated Zn (prepd. from 65 g. Zn wool, allowed to stand 1 hr. under 200 cc. of 5% HgCl_2) boiled 0.5 hr. with 50 cc. of a mixt. of equal parts fuming HCl and H_2O , then treated with the same amt. of HCl and 3 times more with 50-cc. portions of fuming HCl (total length of boiling, 7 hrs.), extd. with Et_2O , washed with H_2O , dried with CaCl_2 , distd. and boiled with Na until there was no further reaction), b. $157-60^\circ$, d_{20}^{20} 0.9700, d_4^{20} 0.9683, n_D^{20} 1.5048, M_D^{20} 38.61, Σ_D^{20} 30.62, $n_F - n_C$ 0.01447; 5 g. with 4 g. AcCl in 25 g. CS_2 added to 6 g. AlCl_3 and 50 g. CS_2 gives 5.5 g. 2-propyl-5-acetothienone, light yellow liquid of aromatic odor, h. $257-60^\circ$, n_D^{20} 1.5438; oxime, m. 55° ; *p*-nitrophenylhydrazones, m. 206° , is brown-red. 3-Isopropylthiophene (Thiele, *Ann.* 267, 133(1892)), b. $153-8^\circ$, d_{20}^{20} 0.9750, d_4^{20} 0.9733, n_D^{20} 1.5052, M_D^{20} 38.44, Σ_D^{20} 30.48, $n_F - n_C$ 0.01409; 3 g. treated by Thiele's method for the prepn. of the acetothienone gave a product which under 15 mm. yielded 0.5 g. b. up to 115° , 1.7 g. b. $115-21^\circ$, 1.2 g. b. $122-8^\circ$ and 0.3 g. residue, n_D^{20} 1.5392. Although the 3 fractions showed the same values for n , that the product is a mixt. of the possible 3-isopropyl-2- and -5-acetothienones is indicated by the fact that the oxime is obtained only as yellowish, uncrystallizable oily droplets and is evidently not homogeneous; the *p*-nitrophenylhydrazones seps. from alc. in brownish yellow needles, m. 141° . Not enough of the material was available for a sepn. of the isomers. Triethyl pentane- α,β,γ -tricarboxylate (58.5 g. from 97.4 g. $\text{PrCH}(\text{CO}_2\text{Et})_2$ added to 11.1 g. Na in 200 g. hot alc., then slowly treated, boiling hot, with 59.1 g. $\text{ClCH}_2\text{CO}_2\text{Et}$ and boiled 15 hrs.), b. 184° ; dropped in the course of 1.5 hrs. into 51.3 g. KOH in the same amt. of boiling H_2O , boiled 2.5 hrs. longer, freed from alc. on the H_2O bath, treated with the amt. of dil. HCl necessary to neutralize the KOH used, extd. with Et_2O , evapd. without drying, slowly heated to 200° until the evolution of CO_2 ceases, treated after cooling with an equal vol. of fuming HCl and satd. with gaseous HCl , it yields 26 g. of propylsuccinic acid as a yellowish white cryst. powder; the Na salt from 26 g. of this acid, distd. with 60 g. P_2S_5 , gives 7.6 g. of 3-propylthiophene, b. $160-2^\circ$, d_{20}^{20} 0.9733, d_4^{20} 0.9716, n_D^{20} 1.5057, M_D^{20} 38.64, Σ_D^{20} 30.56, $n_F - n_C$ 0.01438; gives with phenanthrenequinone an olive-green ring and with isatin- H_2SO_4 a blue color; 4 g. with 20 g. CS_2 and 2.7 g. AcCl gives a product yielding under 22 mm. 1 g. b. up to 123° , 3.7 g. b. $123-7^\circ$, a few drops up to 132° and 0.2 g. residue; it is probably a mixt. of 3-propyl-2- and -5-acetothienones; all fractions show the same value, 1.5428, for n_D^{20} . The oxime seps. as an oil, which when taken up in Et_2O and evapd. leaves an oil solidifying to yellowish white cryst. warts, m. $46-50^\circ$

and yielding a product m. constantly at 108° only after repeated crystn. from ligroin. *p*-Nitrophenylhydrazone, brown-red needles from alc., m. 171° after repeated crystn. The colorless, H₂O-clear propylthiophenes gradually darken in the light on long standing and deposit a small amt. of a dark solid but in the dark there is hardly any perceptible change.

CHAS. A. ROUILLER

Isatin-5-sulfonic acid. J. MARTINET AND O. DORNIER. *Compt. rend.* 172, 330-1 (1921).—Isatin, in 3 parts fuming H₂SO₄ (20% SO₃), heated 30 min. on the H₂O bath, poured on ice, and concd. to a sirup, gave isatin-5-sulfonic acid (A), orange-yellow cryst. powder from EtOAc, m. 143°, very sol. in H₂O and alc. A is identical with the product obtained by Schlieper (*Ann.* 120, 6) on oxidizing indigo carmine, thus proving its structure. The potassium salt forms rosetts of golden yellow needles with 1 H₂O; barium salt, K₂Cr₂O₇-red needles; a "less hydrated" form, straw-colored, crystals from concd. HCl. The phenylhydrazone of A, yellow needles, m. 270° (decompn.), gives Ba and K salts, the latter readily dyeing wool and silk greenish yellow, and condensing in AcOH with indoxyl to form violet needles of indirubin. The oxime of A, yellow needles, sol. in H₂O, gives a pale yellow silver salt not readily sol. in cold H₂O. B. H. N.

Iminazolisopiperidine (tetrahydro-1,3,5-benzotriazole) and its derivatives. S. FRÄNKEL AND KAROLINE ZEIMER. *Biochem. Z.* 110, 234-44 (1920).—Since Welisch (cf. C. A. 7, 1871) obtained but small amts. of tetrahydro-1,3,5-benzotriazole by his method of procedure F. and L. attacked the problem, first removing the CO₂H radical from histidine by bacterial action, either using putrefied pancreatic material or the Twort bacillus in pure culture as the active reagent. The β-iminazolyethylamine thus obtained when condensed with methylal and concd. HCl yielded the HCl salt of the desired compd. Iminazolisopiperidinecarboxylic acid could not be decarboxylated by these means, from which it appears that the bacteria which can easily split off an ω-CO₂H group, are unable to remove the CO₂H attached to a nuclear C. For purposes of identification the picrate, picrolonate, Au, Pt and Hg salts were prepd. and analyzed. They are easily crystd. compds. Acetylation of the substance gave only 1 mono-Ac deriv., benzoylation a di-Bz deriv. On the basis of these results and allied information the structural formula of the HCl salt is given as



The base itself was not prepd. in sufficient amts. for analysis.

F. S. HAMMETT

New method for the preparation of alkamines. II. JIRO TAKEDA AND SAJURO KURODA. *J. Pharm. Soc. Japan*, No. 467, 1-76 (1921); cf. C. A. 14, 179.—T. and K. showed in Paper I that anethole dibromide reacts with urea and goes over to an alkamine by treatment with alkali, and recommended it as a new method of prepn. of the alkamines that have an NH₂ and OH group in α- and β-positions in a side chain of the Ph ring. In the present paper T. and K. report that by previous acetylation, the oxazolidine ring can be broken up much more easily than by the method reported in Paper I. An application of this new method is made to other alkamines and their derivs. (1) Formula for ψ-urea derivs. In the earlier work they used an oxazoline formula for anethole-ψ-urea, but since from the study of the monomethyloxazoline of anethole, isosafrole and methylisoeugenol, they found that the first Me group is attached to N in the ring, γ , ϵ , R'CH.CHR.NMe.C(:NH).O they decided that the oxazolidine formula should be

used instead; thus for anethole-, isosafrole- and methylisoeugenoloxazolidine, is given the structure $\text{MeCH.CHR.NH.C(:NH).O}$ (R = MeOC₆H₄, CH₃O₂C₆H₄, (MeO)₂C₆H₃).

(2) New method. When isosafrole-ψ-urea is heated with glacial AcOH and NaOAc, two compds. are obtained: C₁₁H₁₆O₄N₂ (A) and C₁₁H₁₄O₃N (B), A in larger quantities than

from anethole- ψ -urea, and B, which is acetyl- β -methylenedioxyphenol- α -methyloxazolidine, in traces. A must be acetylisosafole- ψ -urea acetate, but its exact structure was not ascertained. A, when treated with alkali, goes over easily to an alkamine, in spite of the fact that the original isosafole- ψ -urea produces a trace of alkamine under high pressure at a high temp. In this method, A and the acetyloxazolidine need not be sepd. before treating with alkali. From methylisoeugenol- ψ -urea, α -*m*, β -dimethoxyphenyl- β -hydroxypropylamine can be prepared easily by the same method. (3) β -Alkamines. When A is melted, it goes over to acetyloxazolidine with loss of CO₂ and a trace of an alkamine (Cu salt, m. 171-3°); the latter is entirely different from the alkamine prepd. by alkaline treatment of A. It has the structure, CH₃O-C₆H₄CH(NH₂)CHMeOH, isomeric with the normal alkamine, and is named β -isosafolealkamine. When A is treated with alkali and CuSO₄, the normal isosafolealkamine seps. out as Cu salt; a trace of the β -alkamine also seps. on standing. Anethole- ψ -urea acetate also gives the normal alkamine (m. 113°), and β -anetholealkamine (m. 80°). (3) Reaction of the new method. When acetylanethole- ψ -urea acetate is placed in alk. soln., it loses AcOH, and gives an Ac deriv. having 2H₂O. Acetylisosafole- ψ -urea acetate gives in a similar way an Ac compd. having one mol. of H₂O. Two steps must be involved in breaking up these ψ -urea derivs. to alkamines: (a) splitting off NH₂ from imino groups outside of the ring, forming oxazolidine; (b) hydrolysis to alkamine. An attempt to identify this intermediate product during treatment with alkali was not successful. But by heating the ψ -urea with H₂O in a sealed tube, T. and K. isolated this, together with a trace of alkamine. This intermediate oxazolidine goes over quant. to alkamine by heating with alkali. From anethole, they obtained the intermediate oxazolidine, MeCH.CHR.NH.CO.O (R = MeOC₆H₄), m. 110-2°,

and from isosafole the corresponding compd. (R = CH₃O-C₆H₄), m. 170-1°. (4) Me derivs. When the Me derivs. of isosafole- or methylisoeugenol- ψ -urea are heated for 10 hours in alk. soln., *N*-Me derivs. of isosafole- and methylisoeugenolalkamine and NH₂ are produced. On further methylation of these by MeI, di-Me derivs. can easily be obtained, which, when treated with alkali, give off MeNH₂, forming the methylalkamine, which is the same as that prepd. from the monomethyl- ψ -urea. From this, T. and K. conclude that the second Me group must go to the C:NH group outside the ring. The methylalkamine can easily be converted to the di-Me deriv. by MeI. (5) Bz deriv. Using Schotten-Baumann's method, T. and K. obtained the Bz deriv. MeCH.CH(C₆H₄O₂CH₃).NBz.CO.O, m. 180-2°, of the oxazolidine from isosafole- ψ -urea,

with loss of the NH group outside of the ring. (6) Thio derivs. The alkamines of anethole and isosafole when treated with CS₂, give oxazoline mercaptan, unlike PhCH(OH)CH₂NH₂, which gives phenylthioazoline mercaptan. (7) Demethylation. In attempts to remove Me or methylene groups from anetholealkamine and isosafolealkamine, T. and K. obtained α - β -hydroxyphenyl- β -hydroxypropylamine, α - β -hydroxyphenyl- β -hydroxypropylmethylamine and α -dihydroxyphenyl- β -hydroxypropylamine. (7) Naphthol derivs. The new method of prepn. of alkamines can be applied directly to C₁₀H₇ derivs. Dihydronaphthalene bromide reacts easily with urea, forming cryst. dihydronaphthalene- ψ -urea, m. 158-9°. This when acetylated produces an acetate, C₁₀H₇O₂N₂, m. 123-4°, which, when treated with alkali gives β -hydroxytetrahydro- β -naphthylamine, m. 107-8°, hydrochloride, m. 210-5°, picrate, m. 191°. Methylhydronaphthalene urea, m. 66-8°. β -Hydroxytetrahydro- β -naphthylmethylamine, m. 85°. β -Hydroxytetrahydro- β -naphthylmethylamine is an oil; its HCl salt m. 224°, picrate m. 180°. Phys. and chem. characteristics of the various derivs. are compared with those of Mannich (cf. C. A. 4, 2635; Bamberger, Ber. 26, 1838(1893); Ann. 288, 131).

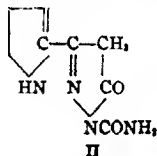
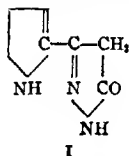
S. T.

Hydrindene. I. W. BORSCHÉ AND M. POMMER. Univ. Göttingen. *Ber.* 54B, 102-10 (1921).—This work was begun in the fall of 1913 but was interrupted by the war, and as the field is being entered from various other quarters the results thus far obtained are published in their present incomplete form. The starting material, hydrindene (A), was prepd. by shaking com. indene, purified by steam distn., in 100-cc. portions with 0.05 g. Pd colloid, suspended in a few cc. of H_2O , with H under atm. pressure for two days. The A, distd. with steam and dried, b. 176° , d_{20} 0.9645, n_D 1.5381, and was free from indene as shown by its behavior towards $KMnO_4$ and concd. H_2SO_4 . Of this, 24-g. portions, with a little red P, were slowly treated in diffuse light with 1, 2 and 4 mols. Br, then heated under a reflux until the Br vapors disappeared and distd. with steam. In the first case almost all of the product volatilized also, the slight residue solidifying on cooling to long needles, m. 125° , of a *bromindene* or *bromohydrindene* (found, 39.86% Br); the volatile part contained, besides unchanged A and a small amt. of a brominated substance, chiefly indene. With 2 mols. Br the proportion of the product not volatile with steam was greater; it only partly solidified on cooling; under 14 mm. it gave up to 120° a fraction of approx. the same compn. as the distillate in the first expt., a chief fraction at $120-60^\circ$, from which the substance m. 125° , sepd. abundantly on standing, and a fraction b. up to 200° from which no well characterized substance has yet been isolated. In the expt. with 4 mols. Br the product contained practically nothing volatile with steam: *in vacuo* it yielded only an insignificant fraction up to 120° , at $120-3^\circ$ a clear yellowish oil, apparently bromohydrindene (found, 4.62% H, 40.68% Br), a fraction b. $125-60^\circ$, consisting chiefly of the compd. m. 125° , and finally, up to about 200° , a heavy red-brown oil. From 20 cc. A, 20 cc. Ac_2O and 2 g. urea nitrate treated dropwise at -10° with 20 cc. HNO_3 (d. 1.52), allowed to stand some hrs. in the freezing mixt. and dild. with ice H_2O is obtained about 50% of *nitrohydrindene*, yellow liquid of characteristic sweetish odor; in aq. suspension with Pd colloid it slowly absorbs H and goes over into the *amine*, b. 241° . The expts. were not carried out further because it was found that the crude NO_2 product contained both the α - and the β -comps. and these could not be sepd. either before or after the reduction. In the sulfonation of A, although it was not found possible to increase the yield of the α -sulfonic acid by changing the temp. it was discovered that the amt. of the β -acid increases steadily with increase in temp. at the expense of the α -acid; to prep. the β -acid, 10 cc. A and 10 cc. concd. H_2SO_4 are heated to 150° with frequent shaking until there is formed a homogeneous dark red-brown liquid; after cooling it is dild. with 50 cc. H_2O , neutralized in the cold with concd. NaOH and satd. with NaCl, whereupon the hydrated Na salt of the β -acid seps.; this is converted through the chloride, m. 40° , into the amide, m. 134.5° , *anilide*, needles from dil. $AcOH$ or alc., m. 129° , and *p-toluidide*, crystals from alc., m. $143-4^\circ$. *Hydrindene- β -sulfonic acid* m. $71-2^\circ$. *Quinol β -hydrindene sulfone*, crystals from dil. MeOH, m. $200-1^\circ$, quickly turns brown in the air. *β -Sulphydrylhydrindene*, yellowish oil, b.₁₄ $254-5^\circ$. *β , β' (?)*-*Dihydrindyl sulfone*, greenish yellow crystals from alc., m. $180-1^\circ$. Attempts to convert the Na salt (14 g.) of the β - SO_3H acid into *β -cyanohydrindene* by distg. with 4 g. KCN gave chiefly A but the nitrile can be obtained from A and $BrCN$; b_{14} 105° . From 12 g. A in 100 g. cold CS_2 and 10 g. $AcCl$ slowly treated with 15 g. $AlCl_3$, allowed to stand some time in the cold and then heated on the H_2O bath until the evolution of HCl ceased was obtained acetylhydrindene (B), b_{14} $267-8^\circ$ (C. A. 14, 3659), 5 g. of which shaken to complete soln. with 200 cc. H_2O , 20 g. NaOH and 6 cc. Br yielded hydrindene- β -carboxylic acid, crystals from C_6H_6 , m. 183° (v. B. gives $178-9^\circ$). Two g. of the crude acid in 4% NaOH slowly treated on the H_2O bath with cold satd. $KMnO_4$ yielded both 1, 2, 4- and 1, 2, 3- $C_6H_4(CO_2H)_3$. The B must, therefore, have consisted of both the α - and β -comps. but the cryst. derivs. obtained from it were apparently homogeneous and as their m. ps. differed but slightly from v. B.'s corresponding comps., they probably belonged to the β -series; possibly

the α -isomers are more sol. and remain in the mother liquors or, as the result of the α -position of the acyl to the alicyclic ring and consequent steric hindrance, they are formed only with difficulty and to a very small extent. *Acetylhydrindene semicarbazone*, crystals from alc., m. 230–1°; *oxime*, prisms from alc., m. 119°, converted by the Beckmann rearrangement into the *acetyl derivative*, m. 108°, of β -aminohydrindene, b. 247°, m. 33–4°. *o*-*Bromoacetylhydrindene*, from 8 g. B in 24 cc. AcOH and 8 g. Br, crystals from ligroin, m. 61°, forms with PhNH₂ in alc. the *o*-*anilino compound*, yellowish needles from alc., m. 134°. *p*-*Methoxycinnamylhydrindene*, from 4 g. B and 3.4 g. MeOC₆H₄CHO in 40 cc. alc. allowed to stand overnight with 20 cc. of 20% NaOH, crystals from alc., m. 92–3°. *Benzoylhydrindene*, obtained like B, although analytically pure, m. quite indefinitely above 40° and therefore probably contained some of the α - along with the β -deriv. *Oxime*, crystals from dil. alc., m. 153–4°, is converted in the Beckmann rearrangement into the isomeric *hydrindene- β -carboxanilide*, leaves from alc., m. 126°, and not into *N*-*benzoyl- β -aminohydrindene*, which was prepd. from aminohydrindene and seps. in long needles, m. 137°.

CHAS. A. ROUILLER

New syntheses in the pyrrole group. Pyrrolic ketonic acids and dipyrrolyl ketone. XIII. BERNARDO ODDO. Univ. Pavia. *Gazz. chim. ital.* 50, II, 258–68 (1920).—O. and Moschini (*C. A.* 6, 3425) obtained Et pyrrolylacetate (A), NH₂C₄H₃COCH₂CO₂Et, which is sapond. by dil. KOH and gives a free cryst. acid. A decomps. easily in dil. alkalis to give α -pyrrolyl Me ketone, EtOH and CO₂. The free acid decomps. similarly at the m. p. (95°); boiled in solvents it decomps. more slowly. The aq. soln. of A with AgNO₃ and a drop of NH₃ gives a white ppt., C₄H₃(COCH₂CO₂Et):NAg, while the free acid, capable of giving an enol, gives a green color with FeCl₃. O. has now studied the action of PhNHNH₂, NH₂CONHNH₂, and NH₃ with A, as well as reactions of the free acid with various salts. A reacts easily in EtOH with PhNHNH₂, giving 3-pyrrolyl-1-phenylpyrazolone (I) by first condensing on the CO group with the NH₂ giving the phenylhydrazone and then splitting out EtOH with the NH group. I with a crystal of



NaNO₂ and some drops of dil. HCl gives a garnet-red color which becomes yellow with alkalis and red again with acids, which reaction characterizes pyrazolones. I in alc. with FeCl₃ gives a red color tending to yellow. These reactions prove the presence of the enol of I. With NH₂CONHNH₂, I does not react so easily but also first gives the semicarbazone, which then loses EtOH to give 3-pyrrolylpyrazolone-1-carbamide (II). The behavior of A in forming I and II corresponds with that of AcCH₂CO₂Et. With NH₃ in abs. EtOH in the sealed tube A gives the corresponding *amide* HN:C₄H₃COCH₂CONH₂. Pyrrolylgyoxylic acid (B), HN:C₄H₃COCO₂H, was obtained in poor yields by oxidizing alkyl pyrrolyl ketones as well as dipyrrolyl (*C. A.* 5, 2638; 6, 3425). 91% yields of B were obtained from ClCOCO₂Et and magnesylpyrrole. The free acid of B was obtained by agitating B with 2.5% KOH and extg. with Et₂O after acidifying with cold dil. H₂SO₄. Ciamician and Magnaghi obtained dipyrrolyl ketone but O. got better results with C₄H₇(COCl):NH and magnesylpyrrole. 2 g. A + 1.80 g. PhNHNH₂ and some drops of H₂O were boiled some time and allowed to cryst. The crystals of I purified from AcOEt were slightly red, m. 193°, sol. in EtOH and C₆H₆. I is sol. in cold concd. H₂SO₄ with a garnet-red color. 2 g. A + 2.2 g. NH₂CONHNH₂, HCl, the equiv. amt. of dry Na₂CO₃, and a few drops of H₂O heated 21 hrs. gave needles of *ethyl pyrrolyl-*

acetate semicarbazone, $C_{16}H_{11}O_2N_4$, crystals from lignin, m. 90° , which did not give the $NaNO_2 + HCl$ reaction for pyrazolones. Another product isolated in small amt. from the mother liquor, m. 192° , and was probably II. 1 g. A in the sealed tube for 2.5 hrs. at 150° with 20 cc. $NH_3 \cdot EtOH$ gave crystals of pyrrolethanamide, white pearly microcryst. powder, m. 184° . Pyrrolacetic acid was obtained from A with 2.5% KOH and purifying from C_6H_6 by heating at 45° . It was dissolved in NH_4OH and evapd. *in vacuo*, giving the easily sol. ammonium salt. The NH_4 salt soln. gave a white ppt. with satd. cold $BaCl_2$; with $FeCl_3$ a red-brown ppt. which dissolves, giving a dark cherry color; with 20% $K_4Fe(CN)_6$ no ppt.; with 10% $MgSO_4$ a flocky white ppt.; with $HgCl_2$ a white turbidity and then a yellow ppt.; with 10% $Pb(OAc)_2$ a yellowish white ppt. 17.05 g. Mg pyrrole were treated with the equiv. amt. of $ClCOCO_2Et$ (freshly distd. and dild. with an equiv. vol. of anhydrous Et_2O) gradually. When a yellow turbidity was formed the mixt. was cooled with H_2O . Finally the mixt. was heated for 2 hrs. on the H_2O bath. The mixt. was cooled and treated with ice. The acid was neutralized with Na_2CO_3 and the 2 layers sepd. The aq. portion was extd. with Et_2O and the whole Et_2O soln. dried with $CaCl_2$ and the Et_2O evapd. The ext. was kept *in vacuo* 24 hrs. over H_2SO_4 and paraffin without crystg. It was then distd. at 5 mm. and the main portion, a greenish brown liquid, distd. at 214° and on standing *in vacuo* this ethyl ester of B crystd.; tabular crystals from H_2O , m. 44.5° , of which crystallographic measurements are given. B was obtained as stated above as needles from C_6H_6 , m. 113° (decompn.). 2.5 g. pyrrol chloride freshly prepd. were treated in dry Et_2O with 1 equiv. of magnesiumpyrrole in Et_2O and sepd. on standing overnight dipyrrol ketone (C), $(NH \cdot C_4H_7)_2CO$ needles from C_6H_6 , m. $100-1^\circ$. C treated in $EtOH$ with $AgNO_3$ and then some drops of NH_4OH gave a yellow ppt. of $CO(C_4H_7 \cdot NAg)_2$, insol. in H_2O and $EtOH$.

E. J. WITZEMANN

Action of bromovaleric acid on amines. BRUNO EMMERT AND ERNST MEYER. Univ. Würzburg. Ber. 54B, 210-3 (1921); cf. C. A. I, 1413.—As shown in the earlier paper, the simultaneous electrolytic reduction of levulinic acid and $PhNO_2$ in H_2SO_4 gives 1-phenyl-3-methyl-5-pyrrolidone (A). The structure of A has been confirmed by its synthesis from $MeCHBrCH_2CH_2CO_2H$ (B) and $PbNH_2$ and it was found that B reacts in the same way with other aromatic amines, but with NH_3 , aliphatic amines and amides no trace of a pyrrolidone but valerolactone (C) is obtained in every case on distn., the NH_3 having split off HBr from B and the resulting C forming with NH_3 $MeCH(OH)CH_2CH_2CONH_3$, which on distn. again decomp. into C and NH_3 . Believing that in the case of aromatic amines the first product might also be C, which then combines with the amine to a pyrrolidone, C was heated with various aromatic amines at 220° ; at first no reaction took place but on adding HBr or HCl to the mixt. the pyrrolidone was formed at once. The accelerating effect of HBr or HCl is explained if it is assumed that it is not C but the γ -halogenvaleric acid resulting from it by the action of the halogen acid which reacts with the amine to form the pyrrolidone. At the high temp. at which the reaction occurs (around 180°) the equil. $C + \text{halogen acid} \rightleftharpoons \gamma\text{-halogenvaleric acid}$ is, to be sure, far to the left but is quickly established, so it is immaterial whether the starting point is a mixt. of C + halogen acid or the γ -halogenvaleric acid itself. A, b. 319.5° , is obtained in 67% yield from 6.2 g. C, 8 g. $PhNH_2$ and 2.5 g. $PhNH_2 \cdot HCl$ slowly heated to 210° and kept at this temp. 20 min. 1-o-Tolyl-2-methyl-5-pyrrolidone (4.8 g. from 4.8 g. C and 10 g. o-MeC₆H₄NH₂·HCl heated 1.5 hrs. at 210°), b. 318° . 1-p-Tolyl isomer, from 5 g. C and 10 g. p-MeC₆H₄NH₂·HCl or from 14 g. B and 17 g. p-MeC₆H₄NH₂, needles from $EtOH \cdot H_2O$, m. $82-3^\circ$. 1-m-Nitrophenyl analog, from 7.6 g. C fused with 10 g. m-O₂NC₆H₄NH₂ and treated with HCl gas while slowly heated to 180° and kept 2 hrs. at this temp., light yellow needles from Et_2O , m. $54-6^\circ$.

CHAS. A. ROULLER

Compounds of pyridine with the alkali metals. IV. BRUNO EMMERT AND RUDOLF

BUCHERT. Univ. Würzburg. *Ber.* 54B, 204-9(1921); cf. *C. A.* 11, 2784.—In the earlier paper it was shown that on cautious treatment of monopyridine-sodium (A) with H_2O there is formed a mixt. of tetrahydrodipyridyls, for two of which it was established that they are tetrahydro- γ,γ' - and - α,α' -dipyridyl, resp. To explain this transformation and the other properties of A a formula for A was proposed according to which the Na is united by a secondary valence to the N atom of a C_5H_5N mol. and two such nuclei are again united through subsidiary valences, partly in the γ,γ' - and partly in the α,α' -positions. But as the solid A shows all the appearances of homogeneity it is now assigned the structure $Na...NC_5H_4...C_5H_4N...Na$, with the assumption that it may react in the form of different tautomers of this structure. As A is converted into tetrahydro- γ,γ' -dipyridyl (B) by H_2O , it seemed possible that the *N*-alkyl derivs. of B might be obtained by treating A with alkyl halides. It was found that when A is treated with Et_2O solns. of MeI, EtBr or $PhCH_2Cl$ its color slowly disappears and a ppt. of Na halide gradually seps. On evapn. of the Et_2O there remains a sirup which is not homogeneous. On account of its instability the expected *N,N'*-dialkyltetrahydrodipyridyls could not be isolated as such but their presence was shown by their very characteristic properties depending on their dissociation into radicals with quadrivalent N. As shown in an earlier paper, they become blue in alc. in a short time; the color immediately disappears on shaking with air and returns after a time on quiet standing. Moreover, the blue alc. soln. is decolorized by I with formation of alkylpyridinium iodides. The dipyridine-sodium (B) which has already been described is now assigned the structure $Na...NC_5H_4...C_5H_4N...Na.2C_5H_5N$. A can easily add not only C_5H_5N to form B but also other substances like $PhNH_2$ or $PhNMe_2$. Two of the C_5H_5N mols. in B can, therefore, be removed or replaced without markedly altering the properties of the resulting substances. A can possibly add more than 2 mols. C_5H_5N . Thus, when C_5H_5N acts on Na it becomes red and then an opaque deep green with sepn. of the green B; this green soln. does not change in color even on long standing but on filtering, protected from the air, it again gives a clear red soln. containing about 1.5% solute, calcd. as B. This remarkable difference in color between the stable green suspension of B and its true soln. indicates that on dissolving it forms a new compd. by the addition of more C_5H_5N mols. *Anilinomonopyridine-sodium*, $Na...NC_5H_4...C_5H_4N...Na.2PhNH_2$, from A and $PhNH_2$, almost black, loses its $PhNH_2$ at 250° in *vacuo*. *N-Dimethylanilinomonopyridine-sodium*, black-green, reconverted into A at 250° in *vacuo*.

CHAS. A. ROULLER

The action of alkyl nitrates on piperidine. DAVID TEMPLETON GIBSON AND ALEXANDER KILLEN MACBETH. Univ. St. Andrews. *J. Chem. Soc.* 119, 438-442(1921).—The alkyl nitrates, reacting with piperidine in a sealed tube under perfectly anhydrous conditions, yield piperidine nitrate, thin lustrous plates, m. $173-4^\circ$, and the alkyl deriv. of $C_5H_{11}N$. *Butylpiperidine*, h. $170-2^\circ$. *Hydrochloride*; *chloroplatinate*, m. 218° . The same reaction occurs with Et_3NH , $Et_2NH.HNO_3$ and Et_3N being formed. *iso-AmNO_3*, $EtNO_3$, $PrNO_3$, $BuNO_3$, *iso-PrNO_3* and *tert-BuNO_3* were studied. C. J. WEST

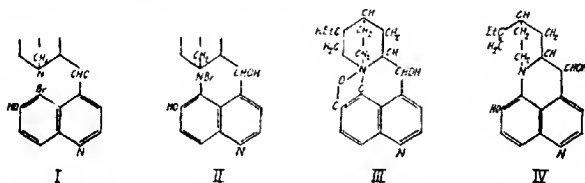
The system, picric acid—5-phenylacridine. HENRY BASSET AND THOMAS ARTHUR SIMMONS. Univ. College, Reading. *J. Chem. Soc.* 119, 416-7(1921).—F. ps. are given for mol. mixts. of the 2 compds. The known picrate of phenylacridine m. 227.7° . A new picrate, $C_{21}H_{19}O_7N_7$ (2 mols. picric acid to 1 of the acridine), was obtained by mixing cold concd. alc. solns. in the proportions of 5 mols. of picric acid to 1 mol. phenylacridine, softened 178° , m. $185-6^\circ$. C. J. WEST

Action of bromine on dihydroquinine and dihydrocupreine. RICHARD WELER. Vereinigten Chinin-Fabriken Zimmer & Co., Frankfurt a. M. *Ber.* 54B, 230-8(1921); cf. Christensen, *C. A.* 10, 47.—*Dihydroquinine dibromide hydrobromide*, $C_{21}H_{21}O_7N_7 \cdot Br_2 \cdot HBr$, is obtained from dihydroquinine-HBr with Br in abs. alc.; the *sulfate* and *hydrochloride* are similarly prepd. *Dihydrocupreine dibromide hydrochloride* and *hydro-*

bromide, $C_{15}H_{21}O_2N_2Br_2 \cdot HX$, are made from dihydrocupreine base and 1 mol. HX , followed by bromination. All these dihydromides are yellow to orange-yellow, well crystd., non-hydrated substances whose m. p. cannot be detd. on account of decompn.; those of dihydroquinine are insol. in alkalis. W. is unable, from the results of his work on the dihydro compds., to confirm C.'s statements that the 2 atoms of ionizable Br in his perbromides of the quinine alkaloids are converted by H_2O into HBr . In studying the action of H_2O on his compds., W. used a method somewhat different from that of C., i. e. he heated them in H_2O in closed bottles in a H_2O bath until they dissolved; on cooling beautifully crystd. mono-Br derivs. sepd., only one of the ionizable Br atoms being converted into HBr and the other entering the C_5H_4 ring of the quinoline group in position 5, as shown by the fact that oxidation with HNO_3 gave cinchoneric acid, thus proving that the Br had not entered the C_8H_5N ring. The dihydromides of both dihydroquinine and dihydrocupreine, on treatment with H_2O and subsequent neutralization, yield the same product, the Me of the MeO group of the quinoline part of the dihydroquinine dihydromides being split off by the treatment with H_2O . Bromodihydrocupreine dihydrobromide, $C_{15}H_{21}O_2N_2Br \cdot 2HBr \cdot 2.5H_2O$, from dihydrocupreine dihydromide hydrobromide, m. 248–53°. Hydrobromide sulfate, $(C_{15}H_{21}O_2N_2Br \cdot HBr)_2 \cdot H_2SO_4 \cdot H_2O$ from dihydroquinine dihydromide sulfate, m. 224–6°. Hydrobromide hydrochloride ($4H_2O$), from dihydroquinine or dihydrocupreine dihydromide hydrochloride, m. 215–8°. Hydrobromide, $C_{15}H_{21}O_2N_2Br \cdot HBr \cdot 3.5H_2O$, from dihydroquinine dihydromide hydrochloride or bromodihydrocupreine dihydromide hydrochloride, m. 91–2°. The action of H_2O on the dihydroquinine dibromides might be explained in the same way as C. explains that of Cl on quinine, viz. that $HBrO$ is first formed from $Br_2 + H_2O$ and adds to the 5-C

atom with elimination of the Me of the MeO group: $MeOC \cdot CH + HBrO = OC \cdot CHBr + MeOH$; as the resulting product does not react with the usual ketone reagents and is

sol. in alkalis the intermediate ketone must have rearranged into the enol $HOC \cdot CBr$. For purposes of comparison the action of Br on *p*-methoxyquinoline was investigated, but contrary to C.'s observations on the action of Cl on this compd., no conversion of the MeO into the HO group occurred. The bromodihydrocupreine salts are well crystd. substances, the acid salts being colored, the neutral salts snow-white (orange-yellow when dehydrated). The m. ps. are not sharp. Although the *o*-position to the HO group is occupied, the salts couple with diazo compds. Addition of NaOAc to their aq. solns. merely salts out the neutral salt; the free bromodihydrocupreine (A), $C_{15}H_{21}O_2N_2Br \cdot H_2O$, is obtained by adding 1 mol. of 0.1 N alc. KOH to the dihydro-



bromide in alc. The Br in the base does not react with $AgNO_3$; when the base is boiled with H_2O until dissolved or heated dry at 120–30° the Br is converted into ionizable Br, probably as a result of a rearrangement of the base into the bromide of an ammonium base (I → II), dihydrocupreonium bromide, which no longer couples with diazo compds. On heating solns. of bromodihydrocupreine salts containing NH_4Cl in NaOH, W. obtained a gray-yellow substance, probably dihydrocupreonium oxide (III), m. 228–30°, which is apparently identical with the product obtained by C. from 5-chloro-6-hydroxy-

cinchonine hydrochloride dihydrochloride and NH_4OH (C. A. 11, 1253); it is almost insol. in alkalis, and in most solvents except acids, gives no ketone reactions, is not reduced by Pd and H, does not decolorize neutral KMnO_4 , gives cinchomeronic acid with HNO_3 , is not affected by boiling KOH and does not couple; it decolorizes acid KMnO_4 , the O bridge probably being broken and a HO group reformed on C atom 6. No salts with dibasic acids could be obtained; those with monobasic acids are in part difficultly crystallizable; all the salts are sol. in alkalis, indicating the presence of a phenolic HO group. *Salicylate*, $\text{C}_{11}\text{H}_9\text{O}_5\text{N}_1\text{HOC}_6\text{H}_4\text{CO}_2\text{H}$, m. 173-5°. *Bromide* (III), from A or from III, seps. with $2\text{H}_2\text{O}$, m. 89-92°. *Chloride*, m. 255-8°. On boiling III with alc. KOH, adding NH_4Cl and crystg. from abs. alc. there are obtained clear crystals, m. 240-2°, sol. in many solvents, especially alkalis, decolorizing KMnO_4 , forming well crystd. salts with mono- and dibasic acids and containing two different HO groups but not giving any ketone reactions or the Fischer toxine reaction with diazobenzenesulfonic acid and Na-Hg. The substance, therefore, probably has the structure IV; efforts to prove the presence of the new double bond by means of PdCl_2 and H were unsuccessful, however. *Chloride*, $\text{C}_{11}\text{H}_9\text{O}_5\text{N}_1\cdot 2\text{HCl}\cdot 3\text{H}_2\text{O}$, m. 98-102°. *Sulfate*, *Oxalate*, m. 203-4°. *Picrate*, m. 159-61°. *Benzoate*, m. 121-5°. CHAS. A. ROUILLER

The relation between crystallographic properties and the constitution of certain organic compounds (SCHLEICHER) 2.

DIELS, O.: Einführung in die organische Chemie. 3rd Ed. Leipzig: J. J. Weber. 327 pp. For review see *Z. angew. Chem.* 1921, Wirtschaftlicher Teil (*Chem. Ind.*), 108.

MALHERBE, D. DU T.: *Organiese Gemie*. Amsterdam: D. B. Centen. 108 pp. f. 2.25. For review see *Pharm. Weekblad* 58, 594(1921).

Biochemical reduction of organic compounds. CARL NEUBERG, AUGUSTE WELDE, NÉE SPITZER, KARL WELDE, ROBERT WELDE, and ELISABETH HEISLER, NÉE WELDE. Ger. 330,812. Mar. 21, 1914. Swelled culture yeast or yeast preps. are mixed with org. substances which are antibodies to culture yeast, and the reaction products are isolated from the mixt. after completion of the reduction.

Hydrogenation of organic compounds. FETTINOUSTRIEGESELLSCHAFT M.B.H. Ger. 329,471, Jan. 23, 1912. (Addition to 312,668.) In place of the compds. mentioned in the principal patent, also other org. compds. capable of hydrogenation are treated at suitable temps. with reducing gases, using metal salts of org. acids, with the exception of the salts of metals of the Pt group.

Hydrogenation of naphthalene. KARL WIMMER. Ger. 300,052, Mar. 14, 1915. Naphthalene is treated with H in heat and under pressure, in presence of a catalyst which is produced by surrounding an org. reducible metal salt in dry state, in the form of a paste or in soln., with a suitable coating, e. g., an oil, and then reducing it. This catalyst is very effective and retains its power of action for a long time.

Hydrogenation of naphthalene. TETRALIN GESELLSCHAFT M.B.H. Ger. 324,861, Feb. 25, 1915. Technical naphthalene is first purified in fused state by stirring with suitable finely divided or porous materials, such as fuller's earth, kieselguhr, animal charcoal, etc., until it no longer shows red coloring with concd. H_2SO_4 , even after standing for some length of time. Naphthalene purified in this manner, mixed in fused state with one of the known active catalyst mixts., is very easily reducible under pressure with the theoretical amt. of H.

Purifying technical naphthalene. TETRALIN GESELLSCHAFT M.B.H. Ger. 324,862, Aug. 1, 1915, and 324,863, May 14, 1916. To purify fused naphthalene in accordance with 324,861 (above), there may also be employed finely divided or readily fusible metals, or metal compds. which contain the metal only loosely bound to a non-acid

residue, such as metal amides, either alone or mixed with the finely divided or porous materials referred to in the principal patent.

Tetrahydronaphthalene. AKTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKATION. Ger. 298,541, Aug. 2, 1916, and additions 298,553 and 301,275, Sep. 13, 1916, and Jan. 19, 1917. Naphthalene vapor is passed, together with H₂, without excess pressure, over a catalyzer of NiO and CuO, or of Mn oxide, NiO and CuO. One of the oxides in the catalyzer may be wholly or partly replaced by an oxide of the rare earths, more particularly Th or Ce oxide.

Producing compounds of acetylene with chlorine. NORSK ALKALI A. S. NORW. 31,377, Nov. 15, 1920. C₂H₂, Cl, or both are dild., before or in the course of the mixing operation or of reaction, with vapors of C₂H₂Cl₄ or other substances produced by the action of C₂H₂ and Cl upon each other.

Chloroform from acetaldehyde. CONSORTIUM FÜR ELEKTROCHEMISCHE INDUSTRIE, G.M.B.H. Norw. 30,949, Aug. 2, 1920. Hypochlorites are made to act, preferably in aq. soln. or suspension, on AcH.

Production of ethylene dichloride. ELECTRIC FURNACE PRODUCTS CO., LTD. NORW. 31,349, Nov. 8, 1920. C₂H₂ in gaseous or liquid state is made to act on liquid Cl.

Formaldehyde from calcium carbonate and hydrogen. ALF SINDING-LARSEN. NORW. 30,801, June 14, 1920. Limestone or marble is heated, while being supplied with H₂, to its dissociation temp. HCHO and Ca(OH)₂ or CaO will then form.

Oxalic acid from carbohydrates. VALENTINER & SCHWARZ, G.M.B.H. Ger. 329,591, Dec. 20, 1913. Carbohydrates are treated, in presence of a catalyzer, with a hydrous mixt. of HNO₃ and H₂SO₄.

Production of three isomeric reduction products of oxycodoinone. FIRMA E. MERCK. NORW. 31,350, Nov. 8, 1920. The oxidation product of thebaine (oxycodoinone), produced, *e. g.*, as described in 286,431 (C. A. 10, 1254), is treated with molecular H in presence of finely divided metals of the Pt group or of colloidal solns. of such metals, or with NaHSO₃ soln. or Zn dust in acetate soln., or with SnCl₂ and HCl. Three reduction products of the empirical compn. C₁₇H₂₁NO₄ will then be obtained.

Manufacture of esters. T. H. DURRANS and W. E. ELLIS. Can. 211,664, May 17, 1921. Esters are made by bringing vapors of an alc. and of a fatty acid into contact with a catalyst carried by a solid body inert to the reaction conditions at a temp. not exceeding 300° the temp. and pressure being such as to maintain the alc., acid and produced esters in the state of vapor without dissociation or decompn. of the compds. The catalyst may be H₂SO₄, H₃PO₄, ZnCl₂, etc., applied to a support such as pumice, coke, asbestos, etc.

Manufacture of esters. ART. A. BACKHAUS. Can. 211,676, May 17, 1921. An org. acid is passed countercurrent to gradually increasing concn. of alc. in the presence of a dehydrating agent. The esters are distd. off.

Manufacture of esters. A. A. BACKHAUS. Can. 211,675, May 17, 1921. An alc. and an acid of the paraffin series are allowed to stand in the presence of a dehydrating agent (H₂SO₄) at normal temp. until the ester seps. from the water layer, which contains the H₂SO₄. The water layer is removed and the oily layer distd. at about 100° until esterification is complete.

Ammonium carbamate. BADISCHE ANILIN- UND SODA-FABRIK. Ger. 294,793, Jan. 23, 1914, and addition 295,389, June 5, 1914. NH₂CO₂NH₄ or mixts. with NH₄ carbonates are obtained by bringing NH₃ and CO₂ together under pressure. For this purpose the CO₂ may be used wholly or partly in the form of NH₄HCO₃ or mixts. containing the latter salt.

Urea. BADISCHE ANILIN- UND SODA-FABRIK. Ger. 295,075, May 7, 1914. The formation of urea from CO₂ and NH₃ or NH₂CO₂NH₄ is greatly accelerated by the presence of certain foreign substances acting as catalyzers.

Methyl bromide. GEORG SCHRÖTER. Ger. 330,642, Nov. 25, 1917. Methane or methane-contg. gas mixts. are passed with Br over catalyzers, more particularly Fe at temps. above 200°.

Methanol. I. SZARVASY. Brit. 159,842, Mar. 4, 1921. In the production of MeOH from MeCl and hot alk. solns., corrosion of the metal reaction vessel or parts thereof by free HCl in the gases produced is prevented by keeping these surfaces covered with a film of the alk. liquid; this may be effected by rotating the reaction vessel or by means of mechanical agitators, atomizers, pumps, etc. In an example, MeCl is heated with lime and H₂O in a rotating Fe vessel.

Aromatic carboxylic acid chlorides. BADISCHE ANILIN- UND SODA-FABRIK. Ger. 331,696, July 17, 1914. The corresponding trichlorides are treated, preferably in presence of catalyzers, with warm water in such a manner that 1 mol. of H₂O will act on 1 mol. of trichloride.

Pseudocumene and benzene. ALF SINDING-LARSEN. Norw. 29,319, June 28, 1920. A carbide which in combination with H₂O forms acetylene is treated with acetone.

p-Cymene from terpenes. AKTIEBOLAGET CENTRALLABORATORIUM. Norw. 31,348, Nov. 8, 1920. Terpenes which have the same atom arrangement in the nucleus as p-cymene, or which upon the rupturing of the double ring contained in them show the C framework of p-cymene, are heated with materials which, each by itself or mixed with one another, split off free H from the terpenes without themselves undergoing any change. Materials may also be used, with the exception of halogens, concd. or alc. H₂SO₄ or Et₂SO₄, which combine chemically with H, while the terpenes are passed over them in the form of vapors or are heated with them in liquid form in open or closed vessels.

Trihydroxyisopentane. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. Ger. 309,111, Aug. 12, 1917. The 3-methylbutenol, specified in 288,271 (C. A. 10, 2502) is oxidized, or its hydrochloride or dichloride is saponified.

Chlorotoluenes. SOC. ANON. DES MATIÈRES COLORANTES ET PROD. CHIM. DE ST. DÉNIS and A. R. WAHL. Brit. 159,837, Mar. 3, 1921. o-ClC₆H₄Me is sepd. from its mixt. with p-ClC₆H₄Me by sulfonating the mixt., interrupting the sulfonation before all the o-isomer has been sulfonated, removing the unattacked oil, and hydrolyzing the resulting o-ClC₆H₄MeSO₃H. Similarly, p-ClC₆H₄Me is sepd. from its mixt. until the whole of the o-isomer and a little of the p-isomer have been sulfonated, and then the unattacked p-ClC₆H₄Me is sepd. Examples are given of both processes.

Furfuralacrolein. WALTER KÖNIG and KARL HEY. Ger. 330,358, Nov. 12, 1919. AcH is made to run slowly at low temp. into furfural in the presence of small amts. of alk. condensation agents.

11—BIOLOGICAL CHEMISTRY

HATTIE L. HEFT, EDGAR G. MILLER, JR. and WILLIAM J. GIES

A—GENERAL

FRANK P. UNDERHILL

Studies on the inorganic constituents of milk. I. Chloride in human milk. W. R. SISSON and W. DENIS. Mass. Gen. Hosp. Am. J. Dis. Children 21, 389-400 (1921).—The method of McLean-VanSlyke was employed with some modifications. To 3 cc. of human milk in a 100 cc. erlenmeyer are added 9 cc. of a 1.2% soln. of picric acid contg. 2 cc. of glacial AcOH per l. After 5 min. 3 cc. of the standard AgNO₃-HNO₃ soln. are added. After 10 min. the liquid is poured into a chloride-free filter. To 10 cc. of the clear filtrate are added 2 cc. of the starch indicator and the excess of Ag is then titrated by means of standard KI soln. The results were checked by means of Volhard's titration after removal of the org. matter with fusion. The

agreement was good in most cases. The specimens of milk were obtained in most instances about 3 min. after beginning of the nursing, when the flow of milk had been established. This method was employed after it was shown that no const. differences occurred in the "fore" and "after" milk. The differences while not constant were marked. The number of specimens examd. was 327. The conclusions were that the "salt" content of human milk is subject to great variations during all periods of lactation. Estn. of the Cl content of human milk can be obtained only by examg. specimens from all nursings during the day. The av. Cl concn. of milk from all types of women at all periods of lactation is 58.2 mg. per 100 cc. milk; the av. of the second week of lactation is 52.6 mg. "Normal" mothers giving large amts. of milk, show a minimum variation in the Cl content with an av. of 38.1 mg. The greatest variation in the Cl content of milk is found in specimens from "nervous" women. Diet is not a cause for the Cl variation in human milk. The Cl concn. of milk from women who give large amts. of milk tends after the second week of lactation to be low and conversely the milk from women giving small amts. of milk is high in Cl.

S. AMBERG

A study of enzymes. M. MÜLLMANN. Baku-Baluchany. *Arch. path. Anat.* (Virchow's) 230, 448-60(1921).—Every serum contains the products of protein digestion substances which react with ninhydrin, demonstrable if large enough quantities of serum are used. Practically all sera contain free enzymes which at times digest one organ, at times another. When one tissue is digested one or more others are not affected. Very frequently it happens that an organ of a young individual is well digested, while that of an adult is very little or not at all digested. In brief, sp. enzymes for certain organs are present in every serum. When we take into account the complex process of growth in which millions of different types of cells partake in building up and breaking down, it is obvious that the content of sp. enzymes varies. Such reactions as that of Abderhalden are due not to protective enzymes but to physiologic enzymes. Life processes, function, nutrition and formation consist in a const. building up and breaking down of living mols. through the agency of enzymes. The formation of enzymes is the basis for the building of living mols. Life is a process involving the const. action of enzymes.

E. B. FINX

Newer studies on the structure of the gastric mucosa. P. G. UNNA AND E. T. WISSIG. *Arch. path. Anat.* (Virchow's) 231, 519-39(1921).—The mechanism of formation of gastric juice has seemed for many years to be satisfactorily explained, but the theory which ascribes the function of pepsin formation to the chief cells is open to a number of objections. There are a number of points still to be cleared up which are open for discussion. The main constituent of the chief cells is the granuloplasm, a chem. well known protein, "cell alhumose" briefly termed cytoze. Its presence, in the chief cells and absence in the investing cells constitutes one of the main points of difference between the two types of cells. This substance is probably only in the nature of a protective substance for the gastric juice. By means of reduction staining with KMnO_4 , strongly reducing granules can be demonstrated in the chief cells. These possess a hitherto unknown function. The source of pepsin is still to be detd., possibly outside the gastric glands. There is much to support the contention of Heidenhain that the production of HCl is connected with the investment cells which would then possess the remarkable function of freeing HCl from the NaCl of the lymph by an enzyme action. The newer finding of peculiar eosinophile cells in the stomach of the rat (Y-cells), surrounding the gastric glands opens up a field for investigation as to their distribution in other animals, and their relation to the function of the stomach. These Y-cells are characterized by their sp. granular staining with Giemsa and May-Grünwald stains typical ring appearance of the nuclei, which are best demonstrated by polychrome methylene blue, and the extraordinary resistance of the granules and nucleus to all trypsin soln. The highly astonishing circumstance, that in the immediate neigh-

borbood of the glands which secrete HCl, cells like the Y-cells occur which store a basic protein, demands a biologic investigation of these cells.

E. B. FINK

An experimental investigation into the action of certain electrical treatments on the blood pressure and metabolism. W. BAIN, W. EDGECOMBE, W. SHIRLEY KIDD AND SINCLAIR MILLER. *Lancet* 1921, I, 905-8.

E. B. FINK

Dielectric constants of biochemical substances. R. KELLER. *Biochem. Z.* 115, 134-58(1921).—An extensive critical discussion of the applications of the detn. of the dielec. const. to biochem. problems, from which the general conclusions are that the phenomena usually designated as electrostatic must be sharply differentiated from the galvanic activities. The electrostatic surface charge of the colloids seems to be the strongest factor in the transport of biochem. materials, and is a main factor in the phenomena of vital staining, animal charcoal, metallic colloids, etc. Coehn's dielectric law (*Ann. Physik*, 64, 217(1898)), is fundamentally a law of biochemistry and the dielec. const. of the biocolloids are fundamental properties of living tissue. Moreover, the isoelec. point of the body colloids contains a main dielec. factor and owes its existence only to differences in dielec. const., which in turn are conditioned by chem. compn. The opinion is expressed that the ionic theory has as its working limit corpuscles of about 10^{-7} cm. in diam. Proteins are not considered as acids, but are nearly neutral and do not unite with alkalis with the formation of salts. This naturally leads to the idea that the application of the H-ion concn. hypothesis by the prevailing methods is unjustified. This conception of the surface charges is used to explain not only the transport of the food colloids through the cell, and many types of H_2O movements, but is also involved as the prime mover of biochem. phenomena, and chem. action of protein mols. and other corpuscles of the same order of magnitude is considered relatively rare. The power of chem. combination in relation to dielec. surface attraction seems progressively to diminish with the increase in the size of the mols. and in the large colloid complexes, such as cellulose, becomes almost eliminated.

F. S. HAMMETT

The retention of formaldehyde in the animal body. E. SALKOWSKI. *Biochem. Z.* 115, 159-67(1921).—S. made a preliminary study of the effect of 0.25 and 0.5% Na_2CO_3 on various concns. of HCHO. He found that when such mixts. were heated there apparently takes place a destruction of the HCHO, the degree of which depends largely upon the relative amt. present, since in concns. of 0.3825% some 98% is destroyed, while when 3.825% is present but 27.84% is not recovered. He also detd. that it is impossible to recover all the HCHO added to blood by the usual distn. method and comes to the conclusion that it is no wonder the compd. cannot be found in blood or tissues or excreted into the urine after exptl. administration, since it is quite probably bound by the cells and slowly oxidized.

F. S. HAMMETT

Further contributions to the theory of invertase action. L. MICHAELIS. *Biochem. Z.* 115, 269-81(1921); cf. *C. A.* 15, 1908.—Since each mol. of invertase is able to exert its peculiar action proportional to its concn. whether or not it is free or adsorbed, since invertase when adsorbed on colloidal Fe hydroxide is not resolved by fructose, glucose, mannose, lactose, or an α - or β -methylglucoside but is resolved by sucrose, maltose, raffinose and serum protein, and since the degree of dispersion or colloidality is one of the properties commonly considered, in which the disperse phase contains surface active mols. and internal inactive mols. so that the active mass is not proportional to the mass of the substance, M. is of the opinion that in an invertase soln. the latter conditions do not obtain and that the law of mass action can be applied to its functioning. The activity of invertase adsorbed on colloidal Fe hydroxide is not associated with the resolving ability of sucrose, since the adsorbed invertase is already active before it is resolved. Moreover, the resolving power of the various sugars cannot as yet be said to have reference to their sp. enzyme relation to invertase.

F. S. HAMMETT

An enzyme which brings about union into carbon chains (Carboligase). C. NEUBERG AND J. HIRSCH. *Biochem. Z.* 115, 282-310(1921).—This paper is a report of studies demonstrating the presence in yeast of an enzyme (named *carboligase*) which has the power to cause the union of sep. C complexes in C-chain compds. The authors by enzyme action, were able to prepare an optically active ketone alcohol from equal mols. of acetaldehyde and benzaldehyde, a reaction which is not known to take place under the influence of any simple catalyzer in the realm of pure chemistry. Just as carboxylase is the simplest enzyme which breaks up the C-chain, so is carboligase an agent for the most simple formation of such a structure. Both catalyzers have a rather wide temp. range for their activity. The steps in the study of the reaction leading to the final conclusion were: sepn. of the condensation product of the reaction of equal mols. of acetaldehyde and benzaldehyde during fermentation, recovery of phenylhydrazones, isolation of semicarbazone, prepn. of thiosemicarbazone and obtaining of *p*-nitrophenylazone. The studies on the biochem. nature of carboligase showed its presence in yeast; its activity in the presence of various fermentation substrates; and its activity after sepn. from the living cell. However, it was impossible to obtain any evidence of enzyme reaction in mixts. of carboligase from yeast, acetaldehyde and benzaldehyde when no fermentation was taking place, though when substrates (sugar, etc.) were present the condensation took place.

F. S. HAMMETT

The chemical composition of the silk-worm egg shell. MASAJI TOMITA. *Biochem. Z.* 116, 40-7(1921).—The shells are first cleaned by extg. with weak HCl, washed with H₂O, boiled up several times with 95% EtOH and finally carefully washed with Et₂O and a mixt. of EtOH and Et₂O. The residual material when hydrolyzed with concd. HCl yielded a mixt. from which the amino acids were sepd. in the form of their esters and found in the following % amts.: glycine, 13.72; alanine, 3.80; valine, 0.28; leucine, 1.46; isoleucine, 0.20; proline 2.17; phenylalanine, 0.69; asparaginic acid, 0.37; glutaminic acid, 4.16; serine, 1.10; cystine, 0; tryosine, 11.19; arginine, 0.19; lysine, 0.39; histidine, present.

F. S. HAMMETT

The distribution of urea in the organism. K. L. GAD-ANDRESEN. *Biochem. Z.* 116, 266-302(1921).—These extensive expts. yielded the following observations. The partition coeff. of urea between plasma and blood corpuscles and between Ringer soln. and blood corpuscles ranges from 0.72 to 0.80, while that between plasma and the secretions of the organism is 1. The coeff. of the distribution between plasma and tissues with the exception of adipose tissue could not be detd. on account of technical difficulties. The coeff. between plasma and H₂O-free fat is 0.07. The urea concn. is the same in all tissues and the blood with the exception of adipose tissue, and decreases in the secretions with increased secretory activity. With the exception of sweat there exists an equil. between the urea concn. of the blood and the various secretions of the organism. Whether equil. between the blood and tissue urea content exists cannot be detd. with abs. certainty, but that such a condition exists is considered as highly probable. The NH₄OH content of the blood, tissues and secretions is the same. Tissues from dogs, rats and frogs were used in the expts. and milk, sputum, sweat, gastric juice, cerebrospinal fluid, bile, and vitreous humor were the secretions studied.

F. S. HAMMETT

Bromine in animal tissues. A. DAMIENS. *Bull. sci. pharmacol.* 28, 205-24(1921).—Using the method already described (cf. *C. A.* 15, 219, 867). D. gives the results of his detns. of Br in the various tissues of many animals and man. From the tables published it is evident that Br is rather widely distributed in the animal organism.

F. S. HAMMETT

Remarks on the contributions of R. Kochmann (hydrogen sulfide formation from sulfates through the action of feces) and M. Kochmann (magnesium, calcium and barium sulfate antagonism). E. SALKOWSKI. *Biochem. Z.* 116, 191-4(1921).—A

polemical discussion of R. Kochmann (*C. A.* 15, 1731), and M. Kochmann (*C. A.* 15, 1577).

F. S. HAMMETT

Alcoholic fermentation. VIII. Influence of zinc chloride on the alcoholic fermentation of living and killed yeasts. S. KOSTYCHEV AND L. FREY. *Z. physiol. Chem.* 111, 126-31 (1920).—See *C. A.* 14, 1560. IX. Influence of cadmium and zinc salts on the yeast enzymes. S. KOSTYCHEV AND S. SUBKOVA. *Ibid.* 132-40. See *C. A.* 14, 1685.

F. S. HAMMETT

Influence of age on blood pigment. W. KÜSTER. *Z. physiol. Chem.* 109, 117-24 (1920).—See *C. A.* 13, 2683.

Synthesis of aromatic amino acids in the living cell. L. HUGOUNENQ AND GABRIEL FLORENCE. *Bull. soc. chim. biol.* 2, 133-6 (1920); cf. *C. A.* 14, 3439.—*Aspergillus*, the black pigment of *Aspergillus niger*, previously described by Linossier (*Compt. rend.* 112, 489-92, 807-S (1891)) is prepd. by extg. the spores with cold 10% NaOH. The ext., on being neutralized with H_2SO_4 , produces a flocculent, black ppt. of the pigment in a more or less pure condition. After purification, the product forms a shining, amorphous powder, insol. in water and in acids, but very sol. in alkalis. It contains 4.97% of ash, in which were present Fe, S, Zn and C. Elementary analysis gave the percentage figures: C 43.5, H 6.9, N 12.6, P 8.7, Fe 0.455, Ca 0.352, Mg 0.126, S 0.57, O 27.367. It appears to contain an indole ring in the mol.

J. C. S.

Temperature coefficients of the action of sucrase. HANS VON EULER AND INGVAR LAURIN. *Z. physiol. Chem.* 110, 55-92 (1920); cf. *C. A.* 14, 2344.—Detn. of the velocities of inversion by sucrase at pairs of different temp. shows that the const. A of Arrhenius's temp. formula (*C. A.* 14, 2344) varies with the temp. in accordance with the equation $A = 11,400 (1 - 0.009t)$, which is in agreement with the results of Kjeldahl (*Bied. Centr.* 1882, 791) and O'Sullivan and Thompson (*J. Chem. Soc.* 57, 834 (1890)). There is no theoretical basis indicating the independence of the const. A of the temp.; according to Arrhenius (*Z. physik. Chem.* 4, 226-48 (1889)), A represents the heat change of the reaction, and there is no reason to assume that this heat change must always be independent of the temp. On the other hand, the Arrhenius temp. formula agrees well with the exptl. results over a wide range of temp., especially for the inversion of sucrose by acids. On the assumption that the velocity of the enzymic inversion of sucrose is proportional to the concn. of the complex mol., sucrose-enzyme, the temp. coeff. of the inversion is detd. by the following magnitudes: (1) the influence of the temp. on the concn. of the active enzyme; (2) the influence of the temp. on the reactivity of the compd. sucrose-enzyme (and the reactivity of the water); and (3) the alteration of the equil. sucrose: enzyme, with temp. The last of these can be detd. quant. on the basis of Michaelis and Menten's theory (*C. A.* 7, 2232), according to which $[S] [E - \phi] = K_M [\phi]$, where $[S]$ is the concn. of the free substrate, $[E]$ the total molar concn. of the enzyme, $[\phi]$ the concn. of the combined enzyme or of the compd. sucrose-enzyme, and K_M the equil. const. The value of the velocity of inversion is given by $v = C [\phi] = C [E] [S] / ([S] + K_M)$, C being a proportionality factor; influence (2) is expressed in the factor C and influence (3) in K_M . The authors have measured the values of the const. K_M of the equil. between sucrose and enzyme for the temp. interval 0-40°, use being made of methods essentially the same as that of Michaelis and Menton (*loc. cit.*). At 25° and for the optimal acidity ($pH = 4.5$), the value of K_M is found to be 26.10^{-3} , which remains unchanged over the acidity region $pH = 3.35-5.65$; the above authors gave 16.10^{-3} . Three-fold increase of the concn. of the enzyme is without influence on the value of K_M . The equil. consts. of the equil. $[S] [E - \gamma] = K_M \phi$, obtained for the temps. 1°, 15°, 25°, and 39°, show that the equil. is altered approx. by 1% per degree rise of temp.; the values obtained at these temps. agree well with those calcd. with the help of the heat of dissoc. Q , this being found by the equation $Q = -RT^2 \cdot d \log k / dT = -[4.58(\log K_1 - \log K_2) T_1 T_2] / (T_1 - T_2)$ to have the mean value

2000 cal. As regards the great difference between the temp. coeff. for enzymic inversion (9400 at 20°) and acid inversion (25,600 at 20°), the results obtained show that this is not due to abnormal alteration of the equil. enzyme: sucrose, but that it can depend only on increase in concn. of the compd., sucrose-acid, as the temp. rises.

J. C. S.

Taurine from herring flesh and its crystallographic investigation. E. BERNER. *Z. physiol. Chem.* 110, 172-4(1920).—B. has sepd. about 0.003% of taurine from the flesh of the herring (*Clupea harengus*), and has obtained it in exceptionally well developed crystals belonging to the prismatic class of the monoclinic system: $a:b:c = 0.6817:1:0.9073$, $\beta = 93^\circ 47'$.

J. C. S.

Enzymes. IV. Emulsin, cytase, ereptase, and urease in germinated barley. D. MAESTRINI. *Atti accad. Lincei* 29, II, 164-6(1920); cf. *C. A.* 14, 2003; 15, 379.—When treated with 0.3% AcOH soln. germinated barley yields an ext. capable of hydrolyzing amygdalin, the emulsin being present in the suspension of the meal as well as in the filtered ext. No cytase, ereptase, nor urease could be detected in germinated barley.

J. S. C. I.

Physico-chemical studies on bioluminescence. IV. The physical and chemical nature of the luciferase of Cypridina hilgendorfi. S. KANDA. Marine Biol. Lab. Kyushu Imperial Univ. Japan. *Am. J. Physiol.* 55, 1-12(1921); cf. *C. A.* 14, 3727.—Luciferase of *Cypridina hilgendorfi* is not pptd. by HgCl₂, tannic acid, picric acid nor by satn. with NaCl. It is pptd. by phosph. tungstic acid, K₄Fe(CN)₆ + AcOH and by 0.5 satn. with (NH₄)₂SO₄, as well as by acetone and alc. The clear filtrate from pptn. with HgCl₂, containing luciferase, gave positive tests as follows: Biuret, xanthoproteic, Millon's, tryptophan and Molisch. After 60 hrs.' dialysis slight amts. diffuse through parchment. Coagulation occurs at about 60°. By cataphoresis thick materials deposits at each pole, neither having the properties of luciferase. It is not clear whether luciferase is a protein or a gum or a compd. which has both characteristics.

J. F. LYMAN

The presence of hemicellulose-cleaving enzyme in resting seed and the solution of hemicellulose by enzyme of higher animals. AUGUST RIPPEL. *Landw. Versuchsst.* 97, 179-193(1921).—Hemicellulose-splitting enzymes occur only in small amts. in *Lupinus angustifolius*, *Galium aparine* and *Asparagus officinalis*. Digestion of hemicellulose in animal bodies is probably dependent upon several factors. F. M. SCHERTZ

Digestive enzymes of silk-worm. SOJIRO KAWASE, KEIJI SUDA and KAKUJI SAITO. *J. Chem. Soc., Japan* 42, 103-17(1921).—Gastric juice and glycerol ext. of stomach walls of 2 species of silk worms were examd. The worms were starved for 30 min., placed in a Buchner funnel, and exposed to vapor of petroleum oil. During anesthesia by the vapor, the worms throw out the gastric juice, which is collected. Out of 200 worms, 80 cc. of the juice were obtained within 5 hours. The stomach wall is dissected out and extd. with 80% glycerol contg. a few drops of toluene for 2 days at low temp. The results show that they contain trypsin, which hydrolyzes fibrin into primary and secondary albumoses and amino acid and which acts best in weak alk. soln. They contain also amyllopsin, which acts best in neutral or alk. soln. and which splits starch to maltose and achrondextrin. The wall contains invertase, which acts best in neutral or alk. solns. Lipolytic action is very weak for both neutral fat and lower esters. They contain neither oxidase, peroxidase nor tyrosinase.

S. T.

Blood of the silk-worm. SOJIRO KAWASE, KEIJI SUDA and KAKUJI SAITO. *J. Chem. Soc., Japan* 42, 118-30(1921).—The blood of young worms and of pupae of 2 species of silk worms was used. The analysis shows that the blood of young worms is amphoterie in reaction, and that of pupae alk. in reaction; the blood of young worms and of pupae has d_4^{20} 1.027 and contains no thrombase; it is not coagulable even after a long standing. It gives decided color tests for protein, except Millon's. The

presence of reducing sugar, NH_4 salt, urea and uric acid could not be detected. Albumin is the main constituent of the protein. Amino acids also are present. It contains oxidase, tyrosinase, trypsin, diastase, and invertase. S. T.

Method for the determination of the molecular weight of a substance requiring a small quantity of the substance. II. An application to the tissue fluids and juices. YOHEI YAMAGUCHI. *J. Tokyo Chem. Soc.* 41, 729-55(1920).—The method Y. used for the detn. of the mol. wt. of a small quantity of a substance (cf. Part I), is used for estn. of the isoosmotic concn. of various animal fluids and the plant juices. The isoosmotic concn. n is detd. by the depression of f. p. according to $n = \frac{\Delta T}{1.85}$. The

blood of healthy adult gave $n = 0.290$; the juice of potato bug 0.237. In plants, various conditions which alter n are investigated. 150 osmotic concns. of the compressed leaf juice are given for the following plants: *Saxifraga sarmentosa*, *Opuntia* sp., *Vitis vinifera*, *Paulownia tomentosa*, *Begonia* sp., *Camellia japonica*, *Pelargonium* sp., *Bellis perennis*, *Glycine soja*, *Bleia hyacinthina*, *Dahlia variabilis*, *Phaseolus vulgaris*, *Dioscorea japonica*, *Nelumbo nucifera*, *Prunus donarius*, *Falosa japonica*, *Magnolia obovata*, *Chrysanthemum sinense* var. *hortense*, *Deutsia scabra* var. *crenata*, *Podocarpus nageia*, *Cryptomeria japonica*, *Abies firma*, *Euonymus japonica*, *Punica granatum*, *Ginkgo biloba*, *Pinus thunbergii*, *Abies veitchii*, *Populus pyramidalis*, *Picea polita*, *Abies sachalinensis*, *Cedrus libani* var. *deodora*, and *Larix leptolepis*. The general conclusions are: Evergreen leaves, with a few exceptions, have higher osmotic concn. than other leaves. In needle leaves there is a close relationship between osmotic concn. and geographical distribution. The juices are more concd. in winter than summer. Withered leaves have higher n than fresh. Maple leaves when colored (i.e. anthocyan is present), have greater concn. than when green. Leaves of annual plants are more concd. osmotically in fall than in summer. The leaves of *Sedum sieboldii* and *Sedum alboroseum* undergo changes in osmotic concns. during day and night. S. T.

Wool fat (LIFSCHÜTZ) 27. Catalysis. VII. Temperature coefficient of physiological processes (DHAR) 2.

MACFARLANE, J. M.: The causes and course of organic evolution. A study in Bioenergetics. New York; The MacMillan Co. 876 pp.

ZINSSER HANS AND OTTENBERG, REUBEN: A laboratory course in serum study. 2nd Ed. revized. New York: The MacMillan Co. 184 pp. \$1.25.

B—METHODS AND APPARATUS

STANLEY R. BENEDICT

The demonstration of glucuronic acid in human blood by the orcinol and naphthoresorcinol method. ERNST DIEBSCHLAG. Mülheim. *Arch. path. Anat.* (Virchow's) 230, 179-90(1921).—The slight increase obtained by the naphthoresorcinol method does not indicate that there is a definite decrease in glucuronic acid in the blood of diabetics since there is an unknown component running parallel with the increase in blood sugar which interferes with the naphthoresorcinol reaction. Among the unknown components are others which yield a positive reaction with naphthoresorcinol. The color reaction with naphthoresorcinol is, therefore, too uncertain to be used for estg. the quantity of paired glucuronic acids in the blood. On the other hand, the expts. show that the naphthoresorcinol method taken together with the orcinol and phloroglucinol methods serves as a very sensitive test. E. B. FINK

The demonstration of occult blood in the feces. ERICH AOLER. *Arch. Verdauungs-Krankh.* 27, 153-90(1921).—An extensive discussion and exptl. study of the problem stated in the title. A. advocates as an essential for proper procedure the use of a diet for a preliminary period of 9 days in which all traces of hemoglobin-contg. foods have been eliminated. Food contg. chlorophyll does not influence the method used. The

simplest method is the reaction with 0.5% benzidine; a comparative test with guaiacol is indicated in determinate cases, though the spectroscopic examn. for pyridine-hemochromogen in the fecal ext. is acknowledged to be the most reliable. F. S. HAMMETT.

New methods for preparation of nucleic acids. R. FRULGEN. *Z. physiol. Chem.* 111, 257-72(1920); cf. *C. A.* 14, 2162.—The nucleoprotein from pancreas was prepd. by digesting the minced organ with hot water, treating the aq. ext. with NaOH, filtering and pptg. with 96% alc. after neutralizing with the AcOH. The nucleoprotein was hydrolyzed with NaOH and mixed nucleic acids were pptd. with 96% alc. These were purified by pptn. with alc. in alk. soln. The two nucleic acids were sepd. by salting out the Na salt of guanylic acid with NaOAc. The tertiary salt of guanylic acid was prepd. by treating the secondary Na salt of the acid with NaOH and pptg. with 96% alc. J. S. C. I.

New method of cleavage for nucleic acids. H. STEUDEL AND E. PEISER. *Z. physiol. Chem.* 111, 297-303(1920); cf. *C. A.* 14, 2161.—Na nucleate from the sperm of the herring was hydrolyzed with $\text{Ca}(\text{HSO}_4)_2$ soln. prepd. by passing SO_2 through a 3% CaCl_2 soln. at $120-30^\circ$ for 2 hrs. The resulting products of hydrolysis were pptd. with Ca acetate and purified by various pptns. From the P and N detns. it was found that the substance thus isolated was identical with thymic acid. On hydrolysis with H_2SO_4 under a reflux condenser thymine was obtained from it. The above method affords a means of prepg. thymic acid with comparative ease, and the acid thus obtained shows great stability. After more than a year it retained its white color, soly. in water, and power of reducing Fehling soln. J. S. C. I.

C—BACTERIOLOGY

A. K. BALLS

The decomposition of pyrrolic acid by various molds. T. NAGAYAMA. *Biochem. Z.* 116, 303-6(1921).—N. tested the effects of cultures of *Monilia candida*, *Oidium lactis*, *Aspergillus niger mutante*, *Mucor plumbeus*, *Mucor rouxii*, and *Mucor racemosus* on pyrrolic acid in the presence of CaCO_3 , Na_2SO_4 , or CaSO_4 . Best results were obtained in the presence of the last 2 compds., which are capable of fixing the aldehyde produced. It was found that all the organisms studied were able to split pyrrolic acid and in the presence of the sulfites acetaldehyde was usually produced. F. S. H.

Influence of temperature on various functions of yeast. H. ZIKES. *Z. ges. Brauw.* 43, 211-3, 316-4(1920); *Centr. Bakt.* 11, 49, 353(1919); 50, 385(1920); cf. *C. A.* 14, 1694.—Z. deals with the influence of temp. on the reproduction, sporulation, fermentation, and liquefaction of yeast, on the shape of cells and the production of fat, glycogen, and pigments, on surface films and giant colonies, on the activity of yeast enzymes, and the transformation of bottom, into top-fermentation yeast. The exercise of some of these functions, e. g., reproduction, fermentation, and sporulation, is influenced at least for a time by the temp. to which the yeast has previously been accustomed. Production of acid, esters and fat is more rapid at high temps. ($20-30^\circ$), than at low ones ($12-15^\circ$), while the converse applies to the formation of coloring matters by pigment yeasts. Low temps. favor the formation of elongated cells, which remain attached, while high temps. tend to produce round or ovoid cells, which sep. more readily. J. S. C. I.

Use of iodine for disinfecting the skin. J. SEDORF. *Act. Chirurg. Skand.* 52, 436-83(1920).—In expts. upon the sterilizing action of I upon liquid cultures of *S. pyogenes aureus* and tetanus bacilli, the best results were obtained by the use of a soln. of I (up to a max. of 1%) in 50% propyl alc. A soln. of I in 96% alc. also gave good results while aq. and ethereal solns. were nearly as effective, but a soln. in benzene had only a slight sterilizing action. J. S. C. I.

Alcoholic fermentation. X. Fermentation is life without oxygen. S. KOSTYCHEV

AND PAUL, ELIASBERG. *Z. physiol. Chem.* 111, 141-56(1920); cf. *C. A.* 9, 471; 15, 2106.—Several species of *mucor* were employed for fermenting sugar under aerobic conditions, and the CO_2 produced through direct respiration was estd. It was found that under the exptl. conditions the organisms oxidized the sugar to an extent sufficient to cover their vital energy. J. S. C. I.

Bacteriological diagnosis of the pollution of water (BORNAND) 14.

D—BOTANY

CARL L. ALSBERG

The chemical constituents of green plants. XIII. The basic substances of green plants. H. FRANZEN, A. WAGNER AND A. SCHNEIDER. *Biochem. Z.* 116, 208-14(1921); cf. *C. A.* 15, 1554.—Basic compds. were found to be present in 28 types of plants examd., which leads to the conclusion that these are normal constituents of green plants. In 13 of the types studied the basic constituents consisted largely of NH_3 , so that this compd. or one from which it is easily split is wide-spread in green plants and is apparently a normal constituent thereof. The amts. of the other basic compds. are much smaller.

F. S. HAMMETT

Distribution of lycorine in the N. O. Amaryllidaceae. II. K. GORTER. *Bull. Jard. bot. Buitenzorg.* III 2, 331-4(1920); cf. *C. A.* 14, 3699.—The crystd. alkaloid has been isolated from the bulbs and roots of *Amaryllis belladonna*, L., *Clivia miniata*, Benth., *Cooperia drummondii*, Herb., *Cyrtanthus pallidus*, Sims and *Sprekelia formosissima*, Herb. (yield, 0.1-0.01% and less). It is doubtless identical with "belamarine" from the first-named species and with "amarylline" from the last named, both isolated by Fragner (*Ber.* 24, 1498-1500(1891)), and also with the veratrine-like alkaloid obtained by Molle from the second species (*Jahresb. Pharm.* 1903, 27). J. C. S.

Latex of *Lactarius vellereus*. J. ZELLNER. *Z. physiol. Chem.* 111, 293-6(1920). —The latex of *Lactarius vellereus* consists mostly of water (80.5%) and a mixt. of stearic acid and a resin (14.65%). The latter fraction can be extd. from the dried latex with ether, from which the stearic acid crysts. out on concn. and the resin remains in the mother liquor. Mannitol, dextrose, mineral substances, and proteins make up the rest of the latex. J. S. C. I.

The presence of nitrates in the leaves of trees growing near picric acid works. B. A. BURRELL AND G. W. DOUGLAS. *J. Soc. Chem. Ind.* 40, 60-1T(1921).—The damaged leaves of a number of trees, as the elm, beech, ash and sycamore, which were injured by exposure to fumes of the oxides of N from nitrating plants for picric acid manuf. were found to contain from 0.012 to 0.084% of nitric N. Undamaged trees which were not exposed to the nitric fumes did not contain any traces of nitrates. No trace of nitrites was found in either the damaged or undamaged leaves. W. H. ROSS

Carbon dioxide nutrition of cultivated plants. BORNEMANN. *Fühling's Landw. Ztg.* 70, 1-7(1921).—A general discussion. F. M. SCHERTZ

Carbon dioxide and plants. AUGUST RIPPEL. *Fühling's Landw. Ztg.* 70, 7-11(1921). —A critical review of the book by E. Reinau. Two possibilities are set forth by Reinau: Either the CO_2 assimilation varies according to the law of the minimum (Mitscherlich's), or some other factor is the limiting one. F. M. SCHERTZ

The function of calcium in the nutrition of seedlings. RODNEY H. TRUB. *J. Am. Soc. Agron.* 13, 91-107(1921).—Seedlings were grown in water culture solns. under lab. conditions. Injury resulted to plants grown in pure water, for water represents a partial ionic vacuum to roots and tends to establish an equil. with cell contents by the withdrawal of ions from the plant. Any one pair of ions (salt) will not fully overcome the injurious action, but it is largely overcome by salts yielding the Ca^{++} ion; to a less degree it is overcome by those yielding Mg^{++} ions and but slightly or not at all by those carrying K^+ or Na^+ ions. CaCl_2 , CaCO_3 , and $\text{Ca}(\text{NO}_3)_2$ are most

abundantly absorbed, while CaSO_4 is absorbed by plants which grow on sandy and acid lands. An increase in the number of kinds of nutrient ions present in the soln. increases the absorption of electrolytes. K^+ ions when offered in a simple soln. are neglected but when accompanied by Ca^{++} ions are absorbed. Ca^{++} ions make K^+ physiologically available, while a less striking action by K^+ ions exists for producing absorption of Ca^{++} ions. As the variety of ions present in the soln. is increased the importance of rather sharply marked proportional relations becomes distinctly less than in simpler solns. The presence of a certain minimal quantity of Ca^{++} ions is probably the most striking single chemical condition of the soln. Doubtless, the physiology of the cell is the basis for an understanding of special services performed by the Ca^{++} ion.

F. M. SCHERTZ

Chemistry of higher fungi. XIV. *Lactarius furus* Scopol., *Lactarius pallidus* Pers., and *Polyporus hispidus* Fr. JULIUS ZELLNER. *Monatsh.* 41, 443-53(1920); cf. C. A. 13, 2547.—*Lactarius rufus* Scopol. The petrol. ether ext. consists almost entirely of *lactarinic acid*, $\text{C}_{18}\text{H}_{34}\text{O}_8$, m. 86° . The alc. ext. contained mannitol, dextrose, and basic compds., which were not identified. The aq. ext. contained a small amt. of viscosin and a considerable amt. of carbohydrates of the type of mycetide, besides inorg. matter. *Lactarius pallidus* Pers. The yield of lactarinic acid is about 1% of the air-dry material. The Et_2O ext. contained an ergosterol-like substance and a yellowish brown resin. The alc. ext. contained mannitol (about 10%) and small amt. of dextrose and basic compds. The aq. ext. contained the same complex carbohydrates as above. Upon hydrolysis of the membrane material, dextrose was obtained, also glucosamine. *Polyporus hispidus* Fr. The petrol. ether ext. is yellowish and salve-like. The sapon. no. of the fat is 158.1, and it contains 18.6% unsapon. matter. The Et_2O ext. contained large amts. of resin and some ergosterol-like substances. The alc. ext. gives a small amt. of dextrose, mannitol and choline, but mostly a slightly sol. material which is to be considered as a "phlobaphen;" this is sol. in hot acetone and dil. alc. It forms a yellow, amorphous nearly ash-free powder, which gives a dirty green color with FeCl_3 . The soln. in dil. NH_4OH is pptd. by $\text{Ph}(\text{OAc})_3$, $\text{Cu}(\text{OAc})_2$ and $\text{Ca}(\text{OAc})_2$, FeCl_3 , ZnCl_2 (brown ppt.) AgNO_3 (black, without reduction), less completely by $\text{Mg}(\text{OAc})_2$. The same ppts. result in acetone, but the pptn. is not complete. Bases dissolve the compd. with a reddish brown color, from which soln. acids ppt. it. The KOH melt gives the reactions of pyrocatechol. The aq. ext. contained the usual carbohydrates and mineral matter. From the hydrolysis products of the membrane glucosamine was isolated as the HCl salt, also glucose, pentosans, but no mannose. C. J. WEST

Biochemistry of the mahua flower (FOWLER, *et al.*) 16.

F—NUTRITION

PHILIP B. HAWK

NORMAL.

The food requirements of children. I. Total caloric requirements. I. E. HOLT AND HELEN L. FALES. Rockefeller Inst. and Babies Hospital, New York. *Am. J. Diseases Children* 21, 1-28(1921).—The av. caloric requirement of children is given as about 100 cal. per kg. at an age of 1 year (about 9.5 kg.). For boys it falls to about 80 cal. at 6 years (about 20 kg.) and remains practically const. at this value up to the age of 15 years, the increasing requirements for activity being met by the reduction in basal metabolism. After a wt. of 50 kg. (about 15 years) is reached, the cal. can be reduced rapidly to adult standards (about 48 cal. per kg.). For girls the requirement falls to 76 cal. per kg. at 6 years (about 20 kg.) and continues at this value until the tenth year. Then it rises, with an increase in proportion to growth and activity. The requirement remains at 80 cal. per kg. until growth is complete, then falls rapidly to adult standards, about 44 kg. per kg. The daily caloric requirement of children of

both sexes during adolescence exceeds by nearly 1000 cal. the requirements of the adult man or woman of moderate activity.

S. AMBERG

The fat content of feces of young calves. P. E. HOWE. Rockefeller Inst. *Am. J. Diseases Children* 21, 57-64(1921).—The data show a high fat content of the feces of calves on approx. the third day after birth. This high fat is accompanied by a relatively high percentage of soap. The dry residue formed on the average about 37, 38, 34, 31, 37, 34 and 33% of the moist feces, starting from the first day. The percentage of the total fat was correspondingly 14, 15, 22, 11, 13, 11, and 13% of the total solids. The percentage of soap was 1, 2, 3.5 and 0.4-0.7%. The soap was chiefly Ca stearate.

S. AMBERG

Effect of a ration low in fat-soluble "A" on the tissues of rats. MARGUERITE DAVIS AND JULIA OUTHOUSE. Univ. of Wisconsin. *Am. J. Diseases Children* 21, 307-11 (1921).—Rats were fed on crushed oats, polished rice, skimmed milk *ad libitum* and a limited amt. of cooked potatoes and egg white. Half-grown males grew on this at a rate of 80-90% of the normal for 3 months. After a period of maintenance, they became emaciated and died. Most of the females reared one litter and a few two litters. Nearly all litters were born in the first two months on the ration. All of the young born of animals kept on this ration deficient in the fat-sol. "A" accessory foodstuff showed lesions.

S. AMBERG

Malt soup extract as an antiscorbutic. H. J. GERSTENBERGER. Western Reserve Med. School. *Am. J. Diseases Children* 21, 315-26(1921).—A certain specimen of malt soup ext. possessed antiscorbutic properties. It is supposed that it was made from barley of the proper age and state of germination or from a lot of barley, possessing an unusually high amt. of potential antiscorbutic material.

S. AMBERG

The presence of sugar in the urine of new-born infants before the intake of food. FRANCES MILLIKIN. Sprague Inst., Chicago. *Am. J. Diseases Children* 21, 484-7 (1921).—The urine of 11 new-born infants, collected before any food was taken, contained 1.72-27.95 mg. sugar or 0.027 to 0.166%. The sugar occurs in fermentable and not-fermentable form.

S. AMBERG

The influence of diet on the energy expenditure in work. J. E. ORR AND J. P. KINLOCH. *J. Roy. Army Med. Corps* 36, 81-6(1921).—Expenditure of energy per unit of work performed is influenced by the nature of the preceding meal. Following a high-protein meal the increase due to work is greater than in the preceding post-absorptive state. Following a high-carbohydrate meal the increase is less than in the preceding post-absorptive state. Following a high-fat meal there appears to be a summation of extra energy expenditure due to food and that due to work. It is suggested that the difference in the results obtained in these 3 cases is due to a difference in the mechanism of stimulation involved in the increase of metabolism following the ingestion of protein and of carbohydrate and fat.

A. T. CAMERON

Recent advances in sciences in their relation to practical medicine and the nutritional requirements of the body. F. G. HOPKINS. Cambridge. *Lancet* 1921, 1, 1-7.—A review of the advancement of the science of nutrition from the purely caloric to a recognition of the importance of the accessory food factors and the relation of these to deficiency diseases.

E. B. FINK

Basal metabolism and its clinical measurement. H. G. EARLE AND STRICKLAND GOODALL. London. *Lancet* 1921, 1, 853-4.—A brief review.

E. B. FINK

Similarity of effects produced by absence of vitamins and by exposure to X-rays and radium. W. CRAMEK, A. H. DREW AND J. C. MOTTRAM. London. *Lancet* 1921, 1, 963-4.—The only demonstrable histological change in rats and mice in the final stage of avitaminosis is an extreme reduction of the lymphocytes both in the lymphoid structures and in the blood. This condition of the lymphoid tissue is practically identical with that produced by exposure to the X-rays and radium. These agents, if given in ad-

equate dosage, not only have the same effect on the animals—they lose wt. and die in a state of emaciation—but degenerative changes in the seminiferous tubules of the testis similar to those observed in avitaminosis occur. It is suggested that probably the lymphoid tissue fulfils a more important function in connection with the nutrition of the animal than is generally recognized, and that the effect produced on the general nutritive condition of the animals by these different agencies may be due to their selective action on the lymphoid tissue.

E. B. FINK

Accessory food factors. I. The remarkable food value of various fats. H. ARON AND R. GRALKA. *Biochem. Z.* 115, 188-203(1921).—Prolonged feeding expts. afford further evidence that certain edible fats contain remarkable accessory food factors entirely independent of their caloric value. The action of these substances is of the nature of a facilitation of metabolic assimilation with consequent improved wt. increase and duration of life, which is not found when fat-poor foods or fats of low biological value are fed. Controls demonstrate that it is not a question of the taste of the ingested food, nor is it a matter of difference in amt. of food intake. The detn. of the food ingested showed that a prolonged diet of a fat-poor nature or of one in which the fat had low biological value ultimately brought the animal to a bad condition in spite of high food intake. Margarine is one of the substances producing this deleterious effect. Butter, egg, and cod-liver oil fat all brought about favorable reaction.

F. S. HAMMETT

Methylation in the animal organism. I. Methylation of pyridine in the rabbit. MASAJI TOMITA. *Biochem. Z.* 116, 48-54(1921).—T. gave daily feedings or injections of 0.5 g. pyridine to rabbits over periods of from 6 to 19 days, and detd. the amt., reaction and pyridine excretion of the urine. In the majority of cases small amts. of pyridine were occasionally recovered but the quantities were so small as to lead to the conclusion that this animal can methylate the compd. administered when on a diet of turnips, or tofukara or even after long fasting. **II. The locus of methylation of pyridine in the animal body.** *Ibid* 55.—Expts. are reported in which the urinary excretion of methylpyridine by the frog after the subcutaneous injection of 0.05 g. pyridine acetate was detd. in a series of normal, splenectomized, pancreatectomized and hepatectomized frogs. The results indicate that this animal in the intact condition and after removal of the spleen or pancreas is able to methylate pyridine but not when the liver has been extirpated. The feeding to a splenectomized dog of 0.25 g. pyridine acetate in 5% soln. did not prevent the excretion of methylpyridine, nor did the subcutaneous injection of 0.5 g. of the salt into a castrated male dog fail to yield analogous results. The observations lead to the conclusion that the liver is the locus of methylation of pyridine.

F. S. HAMMETT

Flours as foodstuffs and the question of vitamins. EM. FERROT AND R. LEQOC. *Bull. sci. pharmacol.* 28, 177-91(1921).—A report of the growth of white rats when fed on diets consisting of various brands of com. flours, compared with controls on diets of adequate biol. value. The results indicate that such food materials are deficient in substances compatible with continued health and life.

F. S. HAMMETT

Digestibility of some hydrogenated oils. A. D. HOLMES AND H. J. DEUEL, JR. Office Home Economics, U. S. Dept. Agr. *Am. J. Physiol.* 54, 479-88(1921).—Corn oil, cottonseed oil and peanut oil were hardened to varying degrees by hydrogenation and the digestibility by man of the resulting fats was detd. Digestibility coeffs. for hardened cottonseed oil m. 35°, 38.6° and 46° were resp. 96.8, 95.5 and 94.9%. For hydrogenated peanut oil m. 37°, 39°, 43°, 50° and 52.4° the digestibility coeffs. were 98.1, 95.9, 96.6, 92.0 and 79.0%. For hardened corn oil m. 33°, 43°, and 50° the coeffs. were 94.7, 95.4 and 88.5%. In general digestibility decreased as m. p. increased. All the fats melting below 46° were well utilized.

J. F. LYMAN

The problem of vitamins and avitaminoses. FIL. BOTTAZZI. *Univ. Naples. Bull.*

soc. hyg. 9, 1-19(1921).—A review of the vitamine problem. The properties and sources of vitamins are discussed as well as their effects upon the growth of young, and the health of mature animals. The researches of Rondoni, Rossi, Romoino, Navaro and Pugliese are considered in some detail. The various theories concerning the physiol. action of vitamins are also considered. • CHAS. H. RICHARDSON

ABNORMAL

Studies in chloride metabolism. I. The chloride content of the blood and serum in pathologic states of physiology and digestion. FELIX BOENHREM. Stuttgart. *Z. exp. Med.* 12, 295-301(1921).—If 0.6% of \bar{s} l. of blood is used as the basis of calcn., there are about 30 g. of NaCl in the circulating blood. According to Magnus-Levy, the body of an adult contains at least 100 g. of NaCl. A value of 0.43% was found in a case of carcinoma of the stomach which was probably due to the cachexia, since in other cases of malignancy the values were practically normal. The NaCl content of the blood in hyperacidity was generally increased but normal and low values were also encountered. In cases of diminished gastric secretion the NaCl values were often considerably low but not uncommonly normal or high. The Cl content of the gastric secretion is independent of the blood content within very wide limits. It is only when the body is markedly deficient in Cl that the Cl content of the gastric juice becomes diminished. The following results were obtained in disturbances of the glands of internal secretion: In osteomalacia there is no change in the Cl content of the blood, even when associated with hyperthyroidism. In pernicious anemia there is a marked increase, and despite achylia in one case there was found a value of 0.57% in whole blood. The increase in hypothyroidism is of interest in view of Eppinger's teaching that thyroid ext. leads to mobilization of Cl. The excretion of Cl in hypothyroidism was found to be diminished and in 2 days less than $\frac{2}{3}$ of the NaCl had been excreted in the urine. II. Daily variations of chloride in the blood and urine, with remarks on the pathogenesis of gastric ulcer. *Ibid* 302-16.—With the formation of Cl in metabolism there is immediately an excretion of Cl through the skin and other Cl depots which produces an increase in concn. of Cl in the blood. The excess of Cl in the blood is excreted through the kidneys and partly used to form HCl in the stomach. The Cl absorbed with the food serve to replace those used in the intestinal tract, blood and depots and in the second phase the current is reversed. The kidneys secrete less than before, as shown by the curve of Cl excretion in the urine. In conditions associated with hyperacidity there is a quant. disturbance in these factors as indicated by the NaCl curve of the urine. Whether the hyperacidity results in an ulcer depends upon the predisposition of the stomach and the condition of the gastric glands, which will be more likely to react to Cl when diseased than when normal. Ten g. of NaCl by mouth in a glass of H_2O produces a decrease in Cl content of the blood in about 20 min. when gastric secretion is normal. In gastric disturbances the Cl curve is straight and with marked secretory disturbances there is a reversal of the normal curve, i. e., a rise instead of a fall. III. The influence of the endocrine glands on chloride metabolism. *Ibid* 317-30.—Thyroid ext. in most cases raises the Cl content of the blood. The most marked effect is observed in hypoacidity and achylia. Thymus ext. causes a depression in Cl content of the blood in persons with healthy as well as abnormal stomachs. Adrenaline diminishes blood Cl as well as Cl in the stomach. The effect is transient and due to interference with mobilization of Cl from the depots. Exts. of the anterior lobe of the hypophysis increase the Cl content of both whole blood and serum. Epiglandol has no influence upon Cl metabolism. Ovarian ext. affects the organs concerned in the excretion of Cl, the stomach and kidneys, by depressing the secretion, at the same time causing marked mobilization of Cl in its depots. The effect varies with the conditions present in the stomach, hyper- or hypochlorhydria. The injection of testicular exs. causes an increase in Cl content of the blood. The majority of internal

secretions stimulate Cl as well as Ca metabolism. Adrenaline and thymus depress, while ext. of epiphysis has no effect.

E. B. FINK

An experimental study of avian beri-beri. G. MARSHALL FINDLEY. *J. Path. Bact.* **24**, 175-91(1921).—As contrasted with normal birds, the livers and brains of birds with beri-beri show a decrease in nucleic acid content, which is more marked in the liver. The administration of vitamin B to a bird suffering from beri-beri is followed by the removal of lipoids from the adrenal cortex, an increase of nucleic acid in the brain, the reappearance of the Nissl granules, and the disappearance of the paralytic symptoms. Vitamin B is an essential element in the synthesis of nucleic acid by the body.

JOHN T. MYERS

F—PHYSIOLOGY

ANDREW HUNTER

Some recent advances in the physiology of respiration, renal secretion, and circulation. J. S. HALRANK. *Brit. Med. J.* **1921**, I, 409-13.—A review. A. T. CAMERON

Chemical influence of the active constituents of the ductless glands. E. C. KENDALL. *Surgery, Gynec. and Obstetrics* **32**, 205-9(1921).—A review. A. T. CAMERON

Muscle pigment. HANS GÜNTHER. Leipzig. *Arch. path. Anat.* (Virchow's) **230**, 146-78(1921).—It is quite certain that the pigment of muscles is not identical with hemoglobin. The total detn. of hemoglobin in animals usually includes the muscle pigment. The heart muscle of the frog contains no myoglobin as demonstrated by the spectroscope. Exts. of the heart muscle of the calf showed definite spectroscopic bands. The soln. gave positive guaiac and benzidine reactions and was decolorized by Günther's method. Upon the addition of $K_3Fe(CN)_6$ the soln. turns yellow and the myoglobin bands disappear, while the bands in the red zone remain. A portion of the ext. was treated with glacial acetic acid and shaken with H_2O ; after a time both solns. were much decolorized and showed no absorption bands. Another portion was treated with alk. trypsin for 24 hrs. and pptd. by heat. The brown ppt. gave a brownish yellow soln. with concd. H_2SO_4 and spectroscopically was negative for hematoporphyrin. It is probable that the pigment in muscles has a respiratory function similar to that of hemoglobin. CO poisoning in which oxymyoglobin is changed to CO-myoglobin, pernicious anemia, hemochromatosis, hematoporphyrinemia congenita, paroxysmal hemoglobinuria and ochronosis are some of the pathological conditions associated with changes in myoglobin.

E. B. FINK.

Contribution to the study of cerebral metabolism by the method of partial circulation. IV. **Internal secretion of the liver and morphological variations of the cerebral neurones.** H. RENAULT-CAPART. *Arch. intern. physiol.* **16**, 119-61(1921).—An attempt has been made in this and previous studies. (cf. C. A. **15**, 886 and 1559) to det. some of the physico-chem. factors underlying cerebral effort, and to find some of the elemental substances which influence the physiol. activities of the brain. The results have led to the conclusion that it is necessary for blood to circulate through the liver if cerebral activities are to continue, and hence gave rise to the opinion that the liver affords this facilitation either through its detoxicating processes or because of the elaboration of a specific internal secretion. The latter hypothesis is supported by the evidence. Moreover when hepatic function is arrested there is produced in the cerebral neurones a condition of chromatolysis, which is succeeded by a chromatophile regeneration corresponding with the return of function of the centers when the blood supplying the brain is allowed to pass through the liver. C., therefore, decides that the formation and repair of the chromatophil substance of the neurones is dependent upon the presence in the blood of the thermo-labile principle secreted by the liver. The statement is made: "Cerebral metabolism in its entirety, psychic effort and conscious perception, are directly dependent on an internal secretion of the liver, elaborated as a thermolabile

substance, the action of which seems to be to facilitate the continuous formation of the chromatophile substance of the neurones."

F. S. HAMMETT

Chemical and physicochemical studies of muscles and muscle extract. VI. The extract of the muscle of the Octopus. G. QUAGLIARELLO. *Arch. intern. physiol.* 16, 228-38(1921).—The animals from which the muscle tissue used in these expts. was taken were cooled to -2° in their natural medium, and then eviscerated and the tissues removed for examn. The juice expressed had a sp. gr. of 1.051, a H-ion concn. of 3×10^{-7} or more, a viscosity of 5.09, an elec. cond. of 0.0233 reciprocal ohms, and a surface tension of 0.68, all at 18° . Coagulation occurred at $54-55^{\circ}$. The ext. contained H_2O 85.8%, ash 3.02%, and total N 1.626%. Under the ultramicroscope there could be distinguished innumerable granules (of myosine) identical with those found in similar exts. from mammalian muscle. The high temp. that is necessary before coagulation, instead of indicating a lack of myosine in cephalopod muscle, finds its explanation in the high viscosity of the ext. due to substances not yet identified. Q. considers that there is no fundamental difference between the general constitution of striated muscle of vertebrates and that of invertebrates. **VII. The fats, cholesterol and lipoids of the extract of striated muscle from the dog.** *Ibid.* 239-49. —When an ext. of striated muscle from the dog obtained by use of the hydraulic press is centrifuged for a long time at a high speed, it does not contain any microscopically visible fat, or fat separable by centrifugation. Nevertheless the ext. contains noteworthy amts. of fatty acids, cholesterol and phospholipins. If the ext. is dild. with isotonic saline and heated to 45° the 2 proteins of the ext. sep. and some 90% of the fatty acids and cholesterol are found to be associated with the myosine granules, the former largely as phospholipins. This leads to the conclusion that the fibrils of striated muscle are largely lipid in nature.

F. S. HAMMETT

Studies of the chemical composition of the thyroid gland. EDGARD ZUNZ. *Arch. intern. physiol.* 16, 288-306(1921); cf. C. A. 15, 888.—Z. presents in this article probably the most extensive tabulation of analyses of the thyroid gland so far available. His results show that the thyroid of man weighs on the average between 26 and 30 g. between the ages of 19 and 55 years. The variability, however, is great. The H_2O content also varies within relatively wide limits but approaches a mean of 75 to 76% for all ages in which the wt. of the gland does not pass 60 g. The av. compn. remains relatively const. in the adult in spite of large individual variations observed at all ages. Always there is found more I in the glands obtained from men between 25 and 55 years than in glands from men of 19 to 24. This av. content of I in the fresh or dry gland tends to increase with the diminution in wt. of the thyroid. Per g. of fresh substance the I content averaged 0.56 mg. from 19 to 44 years; it increases gradually from 0.23 mg. in heavy glands to 0.68 mg. in the glands of low wt. There was found no particular difference in the I content of the 2 lobes.

F. S. HAMMETT

A secretion of nuclear origin. Antithrombin. Anticoagulating properties of the nucleic acids. M. DOYON. *Arch. intern. physiol.* 16, 343-62(1921).—Expts. are reported which demonstrate that the antithrombin obtained from the liver, or from the blood after the intravenous injection of peptone also occurs in all the tissues of the body, and that nucleic acids possess the ability to retard or prevent coagulation *in vitro*. The belief is expressed that this property of the nucleic acids is what keeps the blood fluid in many cases of non-coagulation and that for the first time a proof has been afforded of the participation of the nuclei in the phenomena of secretion. F. S. HAMMETT

Studies of blood corpuscle volumes. I. The osmotic properties of blood corpuscles. R. EGE. *Biochem. Z.* 115, 109-33(1921); cf. C. A. 15, 379.—When blood corpuscles are suspended in solns. of salts with univalent anions, they begin to swell and do not immediately reach a const. vol. This swelling proceeds more rapidly in chloride than in nitrate solns. and is quite considerable, amounting to from 10 to 15% of the orig.

inal vol. This phenomenon explains the singular fact that the blood corpuscle vol. is different in isosmotic solns. of chlorides and nitrates. If the vol. is detd. after the swelling is completed no differences in vol. of the corpuscles in the 2 solns. is observed. This swelling is also observed in solns. of nonelectrolytes such as the mono- and dihexoses, and in solns. of bivalent anion salts such as SO_4^{++} and $(\text{COO})_2^{++}$. These properties are interpreted as indicating a permeability of the corpuscular membrane for a number of anions, but that the permeability velocity varies with the anion.

F. S. HAMMETT

Osmotic pressure in blood corpuscles and plasma. II. R. EGE. *Biochem. Z.* 115, 175-87(1921).—After a brief review of the history of the relation between the osmotic pressure of blood corpuscles and blood plasma, E. reports the results of his detns. on these materials, using freezing and thawing, squeezing through a Buchner press, and saponin as methods for obtaining total hemolysis of the red blood corpuscles. By all 3 methods he found a lower f. p. depression in the blood corpuscle ext. than in the plasma. While this indicates that the osmotic equilibria of plasma and corpuscles are not identical, nevertheless the practical and theoretical difficulties attending the detn. of the osmotic pressure, such as the probability of differences in dissociation at the temp. at which the f. p. is detd. from that in force at body temp., and the fact that in all cases pure H_2O does not freeze out, but mixts. of varying concn. of H_2O and other substances do not allow it to be definitely stated that the osmotic pressure of the blood corpuscles is different from that of the plasma.

F. S. HAMMETT

The partition of water added to blood between corpuscles and serum. TAKEO TAKEI. *Biochem. Z.* 115, 220-34(1921).—T. detd. the relative vol. of serum and corpuscles before and after diln. by detg. the dry matter of the 2 components, by the use of the hematocrit, and by refractometric studies. In the latter series a const. temp. of 17.59° was maintained and the protein or added $\text{H}_2\text{O}\%$ was calcd. by the formula: Protein $\%$ = $\frac{1}{2}$ observed $n_D - [100, (100 + \text{added } \text{H}_2\text{O})] \times (0.00277 + 1.33320) \div 0.00172$. The results of the expts. indicate that when H_2O is added to blood *in vitro* in amts. which might be expected physiologically in the body, the serum takes up relatively more than the corpuscles. Only slight differences were found in bloods from rabbits, man and cattle.

F. S. HAMMETT

Do sub-minimal stimuli influence the course of chemical changes in isolated muscle? J. K. PARNAS AND EMILIA LASKA-MINTZ. *Biochem. Z.* 116, 59-70(1921).—With isolated muscles of summer frogs there was detd. as an index of the effect of sub-minimal stimuli the lactic acid production of the material when subjected to single induction shocks. Temps. were rigorously controlled. Results are reported of the effect of continued sub-minimal tetanization. As a whole the study seems to show that these types of stimulation fail to produce any demonstrable chem. alteration in nerve muscle prepu.

F. S. HAMMETT

The carbohydrate metabolism of isolated amphibian muscle. II. J. K. PARNAS. *Biochem. Z.* 116, 71-88(1921).—There is given a preliminary account of modifications of the method of Parnas and Wagner (*C. A.* 8, 2182) for the microdetn. of total carbohydrate in muscle tissue, the details of which cannot be condensed into a brief abstract, although it may be stated that the tabulated results show remarkably good agreement among themselves. By this method it was found that there constantly occurred a decrease in the total carbohydrate content of isolated frog muscle on stimulation with a max. work done ranging from 1.865 to 3.48 cal. and with an efficiency of from 27% to 54.3%. III. The changes in muscles of pancreatectomized animals. *Ibid.* 89-101.—Using pancreatectomized frogs P. detd. the carbohydrate consumption and lactic acid production of resting muscle, of muscle after oxybiotic and anoxybiotic work, and also the O_2 consumption of muscle fatigued under anaerobic conditions and discusses the results in their bearing on the theory of diabetes. Since in none of the

expts. was there any demonstration of a difference in carbohydrate utilization or decomposition by the muscles from the operated animals when compared with unoperated controls, P. considers that muscle is self-sufficient in the mobilization and consumption of its carbohydrates store and that the idea of an exogenous hormone influence on the consumption is without foundation. Carbohydrate consumption is regulated by the activity, the activity by neural influences (tonus not included). However, the carbohydrate store of the liver, which is a secondary prepn. for muscle, is regulated by mechanism in which hormones play a part, and which accordingly indirectly influence in some degree the store of available carbohydrate for the muscles; metabolism in the muscle itself depends on the temp., the time of year and the internal condition of the tissue.

F. S. HAMMETT

The mechanical efficiency of the oxidation processes taking place in isolated amphibian muscle. J. K. PARNAS. *Biochem. Z.* **116**, 102-7 (1921).—P. reports the results of his detns. on the O_2 consumption as related to induced tension in the isolated frog muscle, in an endeavor to find the mechanical efficiency of such preps. A delicate app. is described and pictured for use in these studies. The tabulated results of the 6 expts. show that muscle tissue has an av. efficiency of some 44%, to which value P. attributes a high degree of exactness. Since the efficiency so calcd. is not less than the efficiency as detd. by the detn. of the carbohydrate utilization the conclusion is made that under the usual conditions of oxidation, carbohydrate can be considered as the sole source of energy for contracting muscle.

F. S. HAMMETT

Colorimetric studies of tryptophan. IV. Melanoid formation in acid hydrolysis of proteins and its dependence on the tryptophan complex. OTTO FÜRTH AND FRITZ LIEBEN. *Biochem. Z.* **116**, 224-31 (1921); cf. *C. A.* **15**, 377.—An inquiry as to whether or not the melanin N and tryptophan content of proteins run parallel, embracing the recorded results of the literature and further exptl. studies. The conclusion is reached that the tryptophan content of a given protein is answerable for the melanoidin formation although the parallelism is not exact, since certain by-products of the carbohydrates of humin nature are mixed in the total mass produced by the acid hydrolysis.

F. S. HAMMETT

Gas exchange and mechanical ability of the frog heart. ERY LÖSCHER. *Z. Biol.* **73**, 67-109 (1921); cf. *C. A.* **14**, 1139.—These expts. on the muscle of the frog heart indicate that an increase in O_2 consumption occurs during isotonic contraction of low grade but remains constant when the primary pressure is raised. The max. of the degree of efficiency is in general somewhat higher than is that of other types of muscle contraction.

F. S. HAMMETT

A note on the blood-coagulating and smooth muscle-contracting properties of tissue extracts. C. A. MILLS, GERALD RAAP AND D. E. JACKSON. *Univ. Cincinnati. J. Lab. Clin. Med.* **6**, 374-88 (1921).—When fresh tissue exts., as ground lung, extd. with physiol. saline, are injected into the circulation of dogs, extensive intravascular clotting occurs, blood pressure drops, and the smooth muscle of the uterus and bladder contracts. When exts. are made from lungs which have been dried and extd. with benzene the coagulant property is lost, and the exts. are actively anticoagulant. No clotting occurs on injection, but the bronchioles contract and blood pressure falls. These effects are probably due to a histamine-like substance in the ext. Lung albumins prepd. by pptg. the globulin fraction at its isoelectric point, give reactions for imidazole compounds, do not cause blood clotting, but do cause blood pressure drop and smooth muscle contraction. An active coagulant may be prepd. from the original lung ext. by sepn. at its isoelectric point, which does not give imidazole tests and, therefore, does not contain histamine-like substances. On injection of this substance extensive intravascular clotting occurs, accompanied by profound bronchiole contraction, symptoms strongly suggestive of histamine action. When this substance is rendered non-coagulant by

alk. treatment, it has no appreciable effect on blood pressure or bronchiole contraction. The authors are of the opinion that when blood coagulates, a substance is set free which acts strongly on smooth muscle. This may be histamine or a nearly related substance.

E. R. LONG

The respiratory quotient and its uncertainty. J. A. FRIES. Inst. Animal Nutrition, State College, Pa. *Am. J. Physiol.* 55, 53-64(1921); cf. *C. A.* 14, 3270.—Sources of error in detg. the respiratory quotient may be considerable. They are (1) if based on short-time expts. the wide difference at different intervals through each day, (2) if based wholly on the compn. of inspired and expired air further error is introduced by the production of gases from outside and unknown sources, e. g., CH_4 and CO_2 from microorganisms inhabiting the digestive tract. There is the further uncertainty as to the relative amts. of those fermentation gases which leave the body by way of the lungs and by way of the rectum. A method of analyzing large samples of air for O_2 in use in F's lab. is described. It depends on the removal of the CO_2 by absorption and then the conversion of O_2 to CO_2 by passing the air through a large silica combustion tube containing tested charcoal heated by a short elec. unit. The CO_2 is removed by absorption in KOH and soda-lime and residual gas passed over CuO heated to redness to oxidize any CO to CO_2 . The gas is again passed through absorbers and CO_2 detd. by increase in wt. The method is rather complicated but gives good results.

J. F. LYMAN

Low oxygen effects during rebreathing. E. C. SCHNEIDER AND DOROTHY TRUESDELL. Med. Research Lab., Air Service, U. S. A. *Am. J. Physiol.* 55, 223-57(1921).—A statistical study of 1050 cases taken from the records of the so-called Official Altitude classification examn., U. S. A. In this test the subject rebreathes 52 liters of air, the CO_2 being removed by NaOH. The normal, or usual, response in respiration, pulse rate, systolic, diastolic and pulse pressures are presented by curves. J. F. LYMAN

The physiology of the stomach. 53. The secretory response of the gastric mucous membrane to water and saline solutions. G. F. SUTHERLAND. Univ. Chicago. *Am. J. Physiol.* 55, 258-88(1921).—The intravenous injection of H_2O and salt solns. in dogs causes gastric secretion. It is suggested that this is mainly an instance of the general tissue action controlling hydremia. H_2O introduced into the stomach stimulates gastric secretion more than the same amt. introduced into the small intestine. The gastric glands when in a state of relative activity respond more quickly and completely to a given stimulus than if relatively quiescent.

J. F. LYMAN

ARNETH, JOSEPH: *Die Qualitative Blutlehre*. Vol. I. *Die Leukozyten*. Leipzig: W. Klinkhardt. 430 pp. M 80. For review see *Deut. med. Wochschr.* 47, 484(1921).

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G—PATHOLOGY

H. GIDEON WELLS

Effect of injection of nonspecific protein on diphtheria virulence tests in guinea pigs. HENRIETTA A. CALHOUN. Univ. Iowa. *Am. J. Diseases Children* 21, 108-128(1921).—Normal horse serum contains less than 3 units per cc. of diphtheria antitoxin. Such serum has a certain protective power against diphtheria in the guinea pig. Egg-white also may have some protective power but much less than horse serum. Diphtheria antitoxin is much more efficient than horse serum.

S. AMBERG

Studies in spasmophilia. III. Blood calcium and calcium therapy in older children with Thiemich's sign. L. V. MEYSENBERG. Univ. of Minnesota and College of Physicians and Surgeons, N. Y. *Am. J. Diseases Children* 21, 150-3(1921).—The Ca was detd. by Lyman's method in citrated plasma. Five children (1 to 5 years of age) whose elec. irritability was surely not increased, had 10.7-11.5 mg. Ca per 100 cc. plasma. Twelve children ($\frac{1}{2}$ to 6 years of age), whose anodal irritability was markedly increased had 8.7 to 11.5 mg. Ca per 100 cc. plasma. S. AMBERG

Infantile scurvy following the use of raw certified milk. H. K. FABER. Stanford Univ. *Am. J. Diseases Children* 21, 400-5(1921).—An infant, 10 mo. of age, having been fed nearly since birth certified raw milk, with addition of small amts. of Na citrate, developed scurvy. S. AMBERG

Indicanuria in the new-born. B. E. BONAR. Presbyterian Hosp., Chicago. *Am. J. Diseases Children* 21, 406-9(1921).—Obermayer's reagent was used to test for indicanuria after pptn. of the urine with a weak soln. of Pb acetate and filtering. Of 338 specimens examd. 28 (8.2%) showed positive indican reaction. The reactions occurred most frequently and were more intense on the 3rd, 4th, and 5th day. No reactions were obtained on the 8th to 15th day. Indicanuria was not associated with any pathological findings clinically. S. AMBERG

The histology and interpretation of Virchow's "fatty ulceration" of the aorta. HANNA SCHIRMER KRISCH. Greifswald. *Arch. path. Anat. (Virchow's)* 230, 191-214(1921).—The fat in Virchow's fatty ulceration of the aorta is derived mainly from fatty metamorphosis *in situ*. The possibility of a hematogenous fatty infiltration must be considered however. In a biol. sense, in those cases in which the microscopic picture resembles atheroma, the process is associated with fatty degeneration. In those cases in which the tissue spaces are not filled with fat, the fat in the cells and ground substance has no degenerative significance. Such lesions may heal by regeneration. E. B. FINK

Epi- and pericellular amyloid infiltration. J. TH. PETERS. Utrecht. *Arch. path. Anat. (Virchow's)* 231, 467-79(1921).—In the human adrenal in amyloidosis, there is no evidence for the contention of Schmidt that amyloid is derived from the blood proteins by pptn. produced through the action of cell enzymes. E. B. FINK

A note on the claim that agglutinins are lipoidal in nature. CHARLES KRAMWIEDE AND W. CAREY NOBLE. Dept. of Health, N. Y. *J. Immunol.* 6, 210-4(1921).—Expts. to test the soly. of agglutinins in lipid solvents furnished no evidence to indicate that they are lipoidal in character. In no case was there a transference of agglutinins by the extg. solvent where it could not be demonstrated there was at the same time a transfer of antigenic proteins. In no case were K. and N. able to reduce the titer of extd. sera beyond a reduction which is explainable by the manipulation of the serum. The methods employed were relatively simple. This was desirable in view of destruction of antibody by more complex methods. However, the methods used would suffice to demonstrate the soly. of agglutinins in the solvents used, if they were sol. in these menstrua. E. B. FINK

The relation between the absorption of antibodies and the isolated protein bodies. ROKURO UMEMURA. Tokyo. *J. Immunol.* 6, 205-22(1921).—The following conclusions are drawn: "the rate of absorption depends upon the quantity of protein associated with it. The quantity of protein being the same, the rate of absorption of antibody associated with isolated protein bodies is slower than that of the whole normal serum. There is no difference in the rate of absorption of the antibodies of each isolated protein. Hence there is an advantage in preferring isolated protein bodies, containing a minimum of protein, in the use of antibodies." E. B. FINK

Specificity of anti-organ sera. MOYER S. FLEISHER AND NATALIE ARNSTEIN. St. Louis. *J. Immunol.* 6, 223-34(1921).—It has been possible by various methods to

demonstrate tissue specificity in liver, kidney, spleen, brain, muscle and testicle. The methods of demonstrating this specificity and its degree varies in the different tissues. In some cases the specificity is absolute, in others it is only relative. The difficulty in demonstrating this specificity lies apparently in the extreme complexity of the biological compn. of the tissues, and possibly in the interrelationship existing between various tissues.

E. B. FINK

Multivalent antibody response to multiple antigens. F. M. HUNTOON AND S. H. CRAIG. Mulford Labs. *J. Immunol.* 6, 235-47(1921).—There is nothing in the immunity mechanism itself which precludes the possibility of a very wide multivalent antibody production. The undoubted fact that many animals under multiple antibody immunization at times fail to respond with as great a vol. of production as occurs with single antigens may be explained by other factors such as a depreciation of the general physical condition due to the large amts. of bacterial protein injected. Proper prepn. of such multivalent antigen and proper regulations of dosage will probably show that a multivalency can be obtained sufficiently broad to cover the fields desired with an adequate concn. of antibody.

E. B. FINK

Researches on the biochemistry of germs and other proteins with special reference to the problems of immunity. II. Preparation of detoxicated vaccines. DAVID THOMSON. London. *Lancet* 1921, I, 849-53; cf. *C. A.* 15, 1926.—A method of prep. detoxicated vaccines is described. Non-toxic substances like sheep-corpuscles lead to a greater production of antibodies than toxins. **III. The compound nature of protein antigens and probable biochemical explanation of "collateral immunity" and non-specific vaccine therapy.** *Ibid*—An explanation of collateral or cross immunity is to be found in the compd. nature of antigens. A given bacterium or protein consists of a "mosaic of antigens" since it is composed of a combination of several different substances or fractions, and when injected it will call forth the formation of a mosaic of antibodies toward these different fractions. It has been shown that the gonococcus contains 75% of alkali-sol. substance. This alkali-sol. substance is, therefore, likely to be the dominant antigen. An injection of gonococci will call forth antibodies to the alkali-, acid-, and alc.-sol. fractions, but the dominant antibody is likely to be that directed against the alkali-sol. part.

E. B. FINK

The production of bactericidins by microorganisms. J. W. McLEOD AND P. GOVERN-LOCK. Leeds Univ. *Lancet* 1921, I, 900-3.—A considerable amt. of general evidence indicates that many bacteria produce substances inhibitory to their own growth and that of other bacteria in the course of reproduction. In the case of the pneumococcus it is believed that conclusive evidence has been obtained that such substances are produced and that they are relatively labile substances destroyed by heat at 80-85°. A free supply of O₂ seems to be important for their production and it seems possible that in this fact lies the explanation of the therapeutic value of O₂ administration in pneumonia. The possible explanations of these findings are various. They have considerable theoretical interest in the sense that a class of substances perhaps of the nature of enzymes, which have not yet been seriously investigated, is brought under examn. Further, it may be possible in taking those substances into account to elucidate some pathol. and bacteriol. problems till now obscure, such as the crisis in pneumonia or the predominance of certain types of bacteria in different parts of the alimentary tract.

E. B. FINK

Skin reactions in toxic idiopathies. J. FREEMAN. *Lancet* 1921, I, 935.—Various methods of prep. test substances are described.

E. B. FINK

Glucemia and glucosuria. GEORGE GRAHAM. London. *Lancet* 1921, I, 951-5.—After a carbohydrate meal there is a slight rise in blood sugar which may last for several hrs. It is probable that the excess sugar is stored in the liver, muscles and else-

where and that only a small part is burned immediately. Expts. in which the liver has been cut out of the portal circulation and those in which the liver is seriously damaged in disease, show that the muscles play an important part in the storage of glycogen and it is possible that the "extra sugar" in the blood after a carbohydrate meal is on its way to the muscles. Levulose produces practically no rise in blood sugar after doses of 50 g. The liver is not the chief storehouse for sugar in the body. The complete disappearance of 25-50% of the sugar which has been injected is most remarkable and suggests that the sugar has been converted into another compound which escapes detection. At the present 3 substances are known which play an important part in sugar metabolism: Cohnheim's substance, which is thermostabile, sol. in alc., and inhibited by trypsin, is apparently necessary for the storage of sugar in the muscles. Dakin and Dudley's substance, which is thermolabile, H_2O -sol., unaffected by trypsin, is apparently necessary to prevent the conversion of sugar into lactic acid. Clark's substance, which is thermolabile, H_2O -sol. (and in diabetic animals must pass through the liver as well) is necessary to enable the muscles to burn the sugar. There is evidence to indicate that the diabetic is unable to store sugar readily and that it circulates in the blood for a longer period than usual. This effect may perhaps be ascribed to the absence of Cohnheim's substance in adequate amts. The persistence of hyperglucemia in severe diabetes, in spite of starvation may be due to absence of Dakin's substance in adequate amts. The incomplete burning of sugar in diabetes may be due to deficiency in Clark's substance. The mechanism regulating the amt. of sugar in the blood is a complicated one and involves the action of the glands of internal secretion.

E. B. FINX

A simple quantitative serum reaction for the diagnosis of syphilis and the expression of results in standard units. GEORGE DREYER AND HUGH KINGSLEY WARD. Oxford Univ. *Lancet* 1921, I, 956-61.—A serum reaction for the diagnosis of syphilis which depends upon flocculation and omits the hemolytic system as an indicator is described. The results compare favorably with the Wassermann reaction, are quant. as well as qual. and the reaction can be standardized.

E. B. FINX

The colloids of urine and stalagmones. H. PRIEBAM AND F. EICHENBERGER. *Biochem. Z.* 115, 168-74(1921).—Using the stalagmometer as described by Schemensky (cf. *C. A.* 15, 386), analogous results were obtained in the testing of urine for colloid in various pathol. conditions. It was, however, found that while the dialysis method previously used by P. and G. Harnheiser (*C. A.* 15, 1155) gave high results for diabetic urines, the stalagmometric tests did not confirm the earlier finding. The explanation for the discrepancy is based on the fact of the great diln. of diabetic urine which distorts the stalagmometric values when compared with urines of normal daily vol. This finding militates against the use of the latter method in cases of polyuria, particularly of diabetic origin. Studies are reported of attempts to improve the technic of the stalagmometric method. It was found that urine can be preserved for as long as 2 days without alteration in its dropping count when HgI_2 is added. This holds to a great degree for normal and acidulated urine and urine from which the colloids have been removed. In view of these facts a procedure was developed as follows: the 24-hr. urine is collected, preserved with a bit of HgI_2 and made to a vol. of 2 l. either by diln. or evapn. under reduced pressure. By such treatment stalagmometric detns. on diabetic urine conform to the results obtained by dialysis.

F. S. HAMMETT

Hemolysis and metallic salts. N. WATERMAN. *Biochem. Z.* 116, 165-70(1921).—W. studied the effect of the addition of various concns. of $CaCl_2$, $BaCl_2$, $ZnCl_2$, $CuCl_2$, $CdCl_2$, $HgCl_2$ on the hemolytic action of Et_2O on suspensions of washed red blood corpuscles and the effect of an emulsion of MeOH ext. of pancreas in 10% and 8% concns. and a 2% agar lecithin emulsion, on the pptn. reaction by $CdCl_2$ in various concns. $CaCl_2$, $BaCl_2$ and $HgCl_2$ failed to inhibit hemolysis by the Et_2O , in fact the latter salt

apparently acted as facilitator of the reaction. When $ZnCl_2$, $CuCl_2$ and $CdCl_2$ in the stronger concns. were present hemolysis did not occur. Pptn. of the agar-*lecithin* emulsion did not occur when the weaker concns. of $CaCl_2$ soln. were added at 37° .

F. S. HAMMETT

Studies of the conditions of precipitation of the Wassermann-reaction antigen. (Heart extract). RUDOLF MÜLLER. *Biochem. Z.* 116, 215-23 (1921).—It appears from these studies that certain $NaCl$ solns. can ppt. dissolved lipid from a H_2O -alc. mixt. and that this depends on the relation between the amts. of H_2O and alc. in the liquid. On the other hand lipid in a colloidal soln. obtained by mixing certain amts. of alc. heart ext. and $NaCl$ soln. can be pptd. by the addition of alc. These phenomena are attributed to lowering of the surface tension due to the alc.

F. S. HAMMETT

Colorimetric studies of tryptophan. V. The proteins of immune sera and their tryptophan content. O. FÜRTH AND F. LIEBEN. *Biochem. Z.* 116, 232-6 (1921).—Detns. are recorded of the total protein, total globulin, euglobulin, pseudoglobulin, albumin, and the tryptophan content of the various fractions in normal serum and in serum from diphtheria, dysentery, and tetanus subjects. A significant increase in the total N over the normal in all of the immune sera is shown which is produced at the expense of the pseudoglobulin. However, since the tryptophan content did not react in the same manner there is no foundation for the opinion that the tryptophan complex plays an important role in immunization processes. cf C. A. 15, 2118.

F. S. HAMMETT

A preliminary study of the etiology of osteomalacia in the city of Bombay. H. S. HUTCHINSON AND P. T. PATEL. *Glasgow Med. J.* 95, 241-55 (1921).—A differential study of the incidence and causes of osteomalacia derived from observations on the Mohammedans and Hindus of Bombay. The large preponderance of the disorder among Mohammedan women of the city is correlated with the "purdah" system, which results in lack of fresh air and exercise. There is no evidence to show that a dietetic deficiency is a cause of osteomalacia as the disease is not uncommon among the wealthier classes of the community; while among the poorer classes the deficiency of animal fat in the diet is common to all. There is no reason to believe that child-marriage and prolonged lactation are important factors. The more or less sudden onset with fever in many cases suggests a possible infective condition.

F. S. HAMMETT

Bacterial power of normal human serum toward staphylococcus and streptococcus. S. SAKANE. *Saikingaku Zasshi* 270, 185-96 (1918); *Jap. Med. Literature* 6, 7 (1921).—Normal human serum exerted but slight action on the staphylococcus, no action on the streptococcus. Serum from a tuberculous patient had less than normal bacteriolytic power for staphylococcus, serum from a patient infected with staphylococcus more than normal bacteriolytic power for that microorganism. Serum from a patient with streptococcus infection possessed bactericidal power for streptococci.

J. S. H.

Ice-water bath in complement fixation for the Wassermann reaction—a shortened technic. W. W. DUKE. *Kansas City. J. Lab. Clin. Med.* 6, 393-8 (1921).—The use of an ice-water bath for one hour for complement fixation for the Wassermann test gives as complete a degree of fixation as incubation in the ice box for four hrs.

E. R. LONG

Studies of heterophile antigen and antibody. T. TANIGUCHI. *J. Path. Bact.* 24, 217-40 (1921).—In these expts. the capacity of a heterophile antiserum for pptg. the lipoids of heterophile tissues is quant. parallel to its hemolytic power for sheep cells, and likewise to its complement-fixing power with heterophile lipoids. Also the amt. of ppt. formed is the sum of that due to non-specific and specific combinations. Thus there is strong evidence that all three reactions are due to the same constituent of the antiserum.

JOHN T. MYERS

Surface tension and anaphylactic shock. AUGUSTE LUMIERRE. *Compt. rend.* 172,

544-6(1921); cf. Kopaczewski, *C. A.* 15, 1572.—L. maintains that the cause of shock is principally due to the presence of flocculi in the vessels. Shock produced by the injection of suspensions of BaSO_4 and anaphylactic shock have the same characters, and are accompanied by the same lesions and symptoms. L. W. RIGGS

Urine hemolysis coefficient. A. AMATI. *Poliadico* 28, 287(1921); *J. Am. Med. Assoc.* 76, 1137.—The hemolytic power of normal urine was tested after the addition of graduated amts. of distd. water. Hemolysis does not occur until normal urine has been dild. about 5-fold but in cancer the ratio is reversed, hemolysis occurring with addition of one fifth part of water. In chronic nephritis hemolysis occurs with a much smaller addition of water to the urine than in the case of urine from healthy persons. It is suggested that these facts may aid in the diagnosis of cancer and nephritis in obscure cases. L. W. RIGGS

Resistance to tuberculosis—A non-immunologic chemical factor worthy of consideration. H. J. CORPER, HARRY GAUSS AND O. B. RENSCH. *J. Am. Med. Assoc.* 76, 1216-8(1921); cf. *C. A.* 14, 1847.—Tubes of glycerol agar were inoculated with human tubercle bacilli, stoppered with paraffined cotton plugs, which allowed easy egress of gases, and inserted into the subcutaneous tissue of dogs, with the opening of the tube exposed to the air, or completely buried in the tissues of the abdominal cavity. After 4 to 6 weeks tubes open to the air generally showed a luxuriant growth, but the buried tubes were in every case negative. The paper is largely a review of the work of others. The following summary and conclusions are stated: Humoral antibodies have failed to explain resistance to tuberculosis; the lymphocyte as the important factor is rapidly being discarded; allergic phenomena require further elucidation, and the phagocytic action of the wandering endothelial cell is recognized as important. Both humoral and cellular mechanisms of resistance leave much to be explained. Weber's (1899) empiric reasoning that deficiency of CO_2 in the body favors tuberculosis, while an accumulation retards it, seems to be highly significant and appears to be substantiated by exptl. work thus far conducted. Three % CO_2 inhibits the growth of tubercle bacilli in the test tube, and 15% is tuberculocidal. From the results of cultures of tubercle bacilli buried in the tissues it appears that CO_2 may play a significant role in resistance to tuberculosis. This reopens the consideration of the influence of fatigue, exhaustion, exposure, metabolic diseases, etc., as probable etiologic factors in the causation of tuberculosis in view of their ability to alter the CO_2 content of the body. L. W. RIGGS

Increased amount of uric acid in the blood in the toxemias of pregnancy. J. LEBLE WILLIAMS. *J. Am. Med. Assoc.* 76, 1297-9(1921).—The cases studied were classified in 4 groups: 5 with eclampsia, 13 with preclampsic toxemia, 7 with hyperemesis gravidarum, and 6 normal pregnant women as controls. Urea N, non-protein N, uric acid, and creatinine were detd. for each person and in some cases several detns. were made for the same patient. Taken in order of groups, the urea N averaged in mg. per 100 cc., 17.2, 15.78, 17.2, 12.76; non-protein N 40.1, 31.0, 36.4, 26.08; uric acid 7.84, 3.98, 4.91, 1.94; creatinine 2.67, 1.53, 2.17, 1.38. The increase of uric acid in the pathol. cases is evident. Delivery and recovery are associated with a gradual return of the uric acid of the blood to its normal amt. Toxic vomiting of pregnancy is associated with increase of uric acid in the blood, while nervous or physiologic vomiting is not. It seems possible to differentiate these conditions by quant. estns. of the uric acid of the blood. L. W. RIGGS

Traité de pathologie médicale et de thérapeutique appliquées. Published under the direction of EMILE SERGENT, L. RIBADEAU-DUMAS AND L. BABONNEIX. Vol. XXXII. Radiologie, radio-diagnostic radiothérapie, radiumthérapie by Bécère, Cottenot and Simone Laborde. Paris: Maloine. 30 fr. For review see *Rev. Electrochim.* 15, 47 (1921).

H—PHARMACOLOGY

ALFRED N. RICHARDS

Further progress in the study of the relative efficiency of the different mercurial preparations in the treatment of congenital syphilis in infants and children as determined by a quantitative analysis of the mercury elimination in the urine. W. R. RAMSEY AND D. A. GROEBER. Univ. Minn. *Am. J. Diseases Children* 20, 199-205 (1920).—The elimination of Hg in the urine after inunction with 50% blue ointment begins soon after administration; the max. elimination occurs during the following 3 days and the elimination is fairly complete within 5 days. The amt. of Hg excreted in the urine depends on the amt. used. With 33 1/3 % ointment the elimination begins the second day. With HgCl ointment the elimination is delayed and the total excretion of Hg is much less than with the Hg ointment. With Hg salicylate in oil, injected hypodermically, the maximal elimination occurs in the first 24 hrs. and it lasts 6 to 7 days.

S. AMBERG

Bromine poisoning through mothers' milk. F. VAN DER BOGERT. *Am. J. Diseases Children* 21, 167-169(1921).—A 6-mo.-old nursing had a skin eruption diagnosed as bromide eruption before the history of the mother's medication had been obtained. The mother had been taking a proprietary medicine contg. about 17% bromides. A small amt. of breast milk contained Br in more than traces.

S. AMBERG

The excretion of vuzin. (isooctylhydrocupreine). E. BOECKER. Koch Inst., Berlin. *Deut. med. Wochschr.* 46, 1020-1(1920).—After administration of vuzin dihydrochloride per os only a small portion, at the most about 1.7%, of the alkaloid is recovered in the urine in 48 hrs. Somewhat more may be recovered from the stools, but not sufficient to ascribe the small amts. collected in the urine to lack of absorption. S. AMBERG

Treatment of sprue by massive doses of sodium bicarbonate. A. CASTELLANI. *Brit. Med. J.* 1921, I, 338-9.—This treatment, given by mouth and intravenously, in conjunction with the usual dietetic measures, gives satisfactory results in a large number of cases, especially as regards the intestinal symptoms.

A. T. CAMERON

Notes on one thousand cases of bilharziasis treated by antimony tartrate. F. O. LASPREY AND R. B. COLEMAN. Old Cairo. *Brit. Med. J.* 1921, I, 299-301.—Successful results on a large scale confirm Christopherson's results.

A. T. CAMERON

The action of methylguanidine and guanidine on the isolated frog heart. GEORG ROSENOW. Königshurg. *Z. exp. Med.* 12, 263-8(1921).—The expts. confirm the older results obtained by other methods and show that methylguanidine and guanidine in higher concns. cause the isolated frog heart to stop in diastole. In addition it is shown that this action can be overcome by thorough washing of the heart and that weaker concns. cause an increase in the height of the systole. Expts. on the frog heart do not furnish any evidence for a relation between methylguanidine and guanidine and anaphylactic shock since the concns. necessary to produce marked effects are larger than can result from breaking down in the animal body of parenterally injected protein.

E. B. FINK

The influence of intravenous infusion of hypertonic sodium and calcium chloride solutions on the blood sugar, diuresis and permeability of the kidneys. P. SCHENK. Breslau. *Z. exp. Med.* 12, 269-74(1921); cf. *C. A.* 15, 557.—Intravenous infusion of 100-300 cc. of 10% NaCl soln. reduces the level of blood sugar in man for a period of 3-4 hrs. In the rabbit there is a physiol. disturbance of cation balance resulting in hyperglucemia. Diuresis is reduced, particularly when large quantities of fluid are given at the same time by mouth, but rises within the next 2 days to 800-1000 cc. Glucosuria never occurs. Intravenous CaCl₂ injections reduce blood sugar in a degree varying with the amt. of salt injected. The degree to which the permeability of the kidneys to sugar can be influenced by the injection of large doses of CaCl₂ cannot be

definitely detd. Apparently there is a decrease in permeability, certainly no increase. On the other hand phlorizin diabetes in man can be overcome by administration of CaCl_2 . The anti-diuretic action of large doses of hypophyseal exts. (10 cc. pituitariol subcutaneously) is increased by preliminary injection of CaCl_2 . E. B. FINK

Pharmacologic and physiologic studies on the surviving human stomach. OTTO TEZNER AND MAX TUROLD. Vienna. *Z. exp. Med.* 12, 275-87(1921).—The human stomach, fundus as well as pylorus, possesses an automatic activity for many hrs. after removal from the body. Death of the surviving stomach occurs in an orderly manner which may be influenced in various ways by poisons. The results obtained with surviving pylorus strips agree in the main with those obtained by roentgenologic methods in the living. Morphine increases the rhythmic activity 2-3 times, acetylcholine causes a rise in the irritability and strength of the individual contractions, while baryta increases the frequency of the rhythm as well as the tonus of the musculature. Baryta proved to be a strong stimulant of the ganglia of Auerbach's plexus. Atropine produces a little change in the spontaneous activity of the normal stomach but interferes with the periodic activity of muscles under the influence of acetylcholine. Adrenaline has an inhibitory action on the rhythmic contractions of pylorus strips with the exception of those produced by acetylcholine vagal action. Papaverine interferes with all automatic action. E. B. FINK

Initial inhibition of urinary secretion induced by intravascular injections of hypertonic sucrose solutions. E. WERTHIMER AND CH. DUBOIS. *Arch. intern. physiol.* 16, 307-24(1921).—Using dogs as the exptl. animals it was found that the intravenous injection of hypertonic sucrose solns. often produces as an initial effect an inhibition or slowing of urinary secretion, at a time when the arterial pressure and the kidney vol. are strongly increased. These facts do not coincide with the filtration theory of urine secretion and are explained on the basis of a probable sudden loss of H_2O from the secretory elements of the kidney due to the osmotic pressure of the blood, which has been increased by the sucrose injections. Many tracings are given. F. S. H.

The toxicological test for cytosine. H. FUCHNER AND E. MERTENS. *Biochem. Z.* 115, 262-8(1921).—Because of certain chem. and pharmacol. similarities of cytosine and nicotine a study was made of the reactions of the frog and leech to the drug as a manner of differential testing. It was found that the compd. caused an increase in tone of the latter, as does nicotine, but is relatively but half as strong as the latter. The 2 compds. can also be distinguished by their action on the frog, since cytosine does not produce the peculiar leg effects produced by nicotine, while it does have a marked curare effect, which nicotine does not possess. F. S. HAMMERT

The biological effects of proteinogenous amines. A contribution to the question of the acetonitrile reaction. O. WUTH. *Biochem. Z.* 116, 237-45(1921).—W. found that tyramine and diiodotyramine when administered to mice protected them from doses of acetonitrile ordinarily fatal, thus simulating thyroid in their action. β -Imidazolylethylamine was ineffective. F. S. HAMMERT

The significance of colloidal nutrients for the function of the normal, exhausted and poisoned heart. S. G. ZONDEK. *Biochem. Z.* 116, 246-61(1921).—Z. made colloidal Ringer soln. by the addition to it of gum arabic, gelatin or gum tragacanth in such proportions as to render it isoviscous with blood serum. The results of the expts. with the frog heart showed that the induced colloidal character of the soln. *per se* has no significance for the functioning of this organ. Although colloids such as gelatin and gum arabic raise the tonus of the normal heart, restore that of the exhausted heart and even bring about recovery from certain poisons, the effect is considered as due not to the colloid nature of the fluid, but to its content in inorg. constituents, particularly Ca. Pharmacologically indifferent colloids, which contain but little inorg. salt in the concn. used, are inactive. Sol. starch acts as an org. foodstuff. F. S. HAMMERT

Are the chlorine ions of Ringer solution replaceable by other anions as tested on the beating frog heart? E. R. O. FINCKH. *Biochem. Z.* 116, 262-5(1921).—When the isolated frog heart is treated with Ringer soln. in which NaCl has been replaced by NaBr it continues to beat for a long time, which leads to the conclusion that the Cl ion plays no peculiar role in this connection. When NaI is substituted for the NaCl, harmful effects are observed which are attributed to the formation of elementary I₂. A further test with NaNO₃ gave a similar result and here the toxic effect is explained by the possible formation of NO₂ ions.

F. S. HAMMETT

Experimental studies on the poison of the bee. Miss R. LYSY. *Arch. intern. physiol.* 16, 272-87(1921).—The injection of the poison of the bee into rabbits causes a retardation of blood coagulation, a fall in blood pressure, an increased respiration and an increased intestinal peristalsis, all of which are evidences of a proteotoxic process. The dog reacts to similar treatment by arterial hypertension. L. is of the opinion that the poison is similar to snake venom in its proteotoxic characters.

F. S. HAMMETT

The cardiac action of substances of the quinoline group. Quinoline, quinolidine, lepidine, o-toluquinoline, thalline, p-methoxyquinoline, tetrahydroquinoline. HENRI FREDERICQ AND EMILE F. TERROINE. *Arch. intern. physiol.* 16, 325-42(1921).—The action of these compds. was studied on the artificially perfused isolated heart of the tortoise, various concns. of the substances tested being used. All the compds. studied were in the form of their sulfates and were dissolved in Ringer soln. Extensive protocols are given, which tend to show that these substances have qualitatively a similar action as have quinine, quinidine, cinchonine and cinchonidine. Weak doses diminish either or both the frequency and contractility. They can produce arrhythmia, extra-systole incomplete blocking, etc. Strong doses stop the heart in diastole or half diastole. The presence of a methyl group increases the toxicity, regardless of its location in the mol.; the methoxy group has a similar augmenting effect, as does hydrogenation of the mol.

F. S. HAMMETT

Use of atropine or amyl nitrite in the diagnosis of bradycardia. HENRI FREDERICQ. *Bull. acad. roy. méd. Belg.* [5] 1, 7-12(1921).—Clinically a positive response to these tests does not necessarily mean hypertonicity of the pneumogastric, since a ventricular bradycardia can be produced in the dog by compression of the auricular-ventricular region, giving an accelerated rhythm on intravenous injection of atropine or inhalation of amyl nitrite.

M. HEIDELBERGER

Observations on the pharmacology of some benzyl esters. CARL NIELSEN AND JOHN A. HIGGINS. Abbott Labs., Chicago. *J. Lab. Clin. Med.* 6, 388-92(1921).—Emulsions of benzyl compds. in gum acacia are most favorable for intravenous administration. Marked intestinal relaxation follows the injection of benzyl benzoate and cinnamate, the latter being more active. Both compds. diminish the contractions caused by BaCl₂. The cinnamate lowers blood pressure less than the benzoate. Benzyl cinnamate is inactive when administered orally. The coagulating time of blood is not affected by injection of these drugs.

E. R. LONG

A difference between the mechanism of hyperglucemia produced by ether and by chloroform. E. L. ROSS AND L. H. DAVIS. Northwestern Univ. *Am. J. Physiol.* 54, 474-8(1921); cf. C. A. 15, 1763.—Blood sugar was detd. in dogs (1) after 30 mins. Et₂O anesthesia and after 15 mins. on the following day; and (2) after 30 mins. CHCl₃ anesthesia followed by 15 mins. Et₂O anesthesia on the following day. There was apparently an increased tendency for Et₂O to liberate dextrose the day following 30 mins. of Et₂O anesthesia. The day after 30 mins. CHCl₃ anesthesia, Et₂O anesthesia failed to produce as great a hyperglucemia as in normal animals. It is concluded (1) that Et₂O anesthesia does not produce any injury to dextrose mobilization that can be detected the following day, and (2) that CHCl₃ anesthesia does injure the mechanism of dextrose mobilization according to the degree of injury to the liver cells. J. F. LYMAN

Glucosuric action of atropine. ALESSANDRO ROSSI. *Atti ist. Veneto* 78, 354-7(1919).—Atropine injected into rabbits after abundant feeding, in doses of 0.005-0.015 g. per kg., produced no glucose in the urine, while injections in well fed dogs in doses of 0.00307-0.00671 g. per kg. produced glucosuria in 6 cases out of 10. Large individual differences were noted, small doses causing the appearance of glucose in some cases, while large doses were without effect in others. The action is attributed to a paralyzing effect on the glucoinhibitory nerves and particularly on the vagus. The failure with rabbits is believed due to the strong tolerance of these animals for atropine, and to a probable lack of tone in the branch of the vagus which controls hepatic glucogenesis. M. R. S.

Purgative action of sulfur. A. M. M. VAN DER WILLIGEN. *Arch. ges. Physiol.* 186, 173-84(1921).—The addition of S to the food of cats exerted a marked effect upon digestive processes; the small intestine was filled and emptied somewhat earlier than after the same meal lacking the S. Passage of food from the stomach and the appearance of food in the colon was but slightly changed, but the transportation of material from the proximal to the distal colon was markedly hastened. The observations were made by Röntgen-ray examn. and by autopsy findings. Under normal conditions the chyme entering the proximal colon becomes thickened, whereas under the influence of S, and the H_2S produced, such thickening is prevented or inhibited and progress is more rapid. By injection of H_2S into the proximal colon the period of passage was reduced from 2 1/4 hrs. (with an equal amt. of water injected) to 23 mins. S caused no irritation of the mucosa. G. H. S.

Purgation by calomel. A. M. M. VAN DER WILLIGEN. *Arch. ges. Physiol.* 186, 185-92(1921).—Calomel was given to cats along with their food (200 mg. per kg.). Röntgen-ray examn. showed that in this dosage calomel slightly hastened the discharge of the food from the stomach and markedly stimulated the passage of the material through both the small and large intestines. Peristalsis in the small intestine was increased, and although in the proximal portion of the large intestine antiperistalsis was increased rather than diminished, very powerful peristaltic waves were present. Absorption was not affected. The laxative effect of calomel is due to the stimulation of peristalsis. No fluid secretion is caused and only in the ileum and proximal large intestine is there a slight mucus secretion. G. H. S.

Purgation by phenolphthalein. A. M. M. VAN DER WILLIGEN. *Arch. ges. Physiol.* 186, 193-99(1921).—The oral administration of phenolphthalein to cats (2 g. per kg.) had no effect when given as a single dose, but daily doses of 1/2-1 g. over a period of several days caused no change in the gastric processes, only a very slight change within the small intestine, but hastened the entrance of material into the large intestine and accelerated its passage through the colon. The drug did not affect absorption nor cause abnormal secretion. It slightly stimulated peristalsis and but slightly altered the concn. of the chyme. G. H. S.

MAGNUS, R.: Einfaches pharmakologisches Praktikum für Mediziner. Berlin: Jul. Springer. 51 pp. M. 14 + T.-Z. For review see *Chem.-Zig.* 45, 400(1921).

I—ZOOLOGY

R. A. GORTNER

The formation of *D*-lactic acid in the organism. MASAJI TOMITA. *Biochem. Z.* 116, 1-11(1921).—Using the hen egg as a basis of experimentation T. found that the yolk contains 0.0111% and the white 0.0058% of *D*-lactic acid before incubation, but that during brooding this compd. increases markedly up to the 5th day, after which it decreases to values approaching those found in the fresh material. The increase is more marked in the yolk. These observations lead T. to the opinion that the glucose present in fresh egg is the source of the *D*-lactic acid produced during incubation.

F. S. HAMMETT

The rest-nitrogen content of the white and yolk of hen eggs on incubation. MASAJI TOMIYA. *Biochem. Z.* 116, 12-4(1921).—T. detd. the total non-protein N, the non-protein N pptd. by phosphotungstic acid, and the sol. N not pptd. by phosphotungstic acid which could be detd. according to Van Slyke's method (*C. A.* 7, 3980) in the whites and yolks of freshly fertilized eggs, and in eggs after 1, 3, 7, and 14 days of incubation. The results are presented in the appended table and show a general increase in the constituents detd. Nevertheless the absolute increase, particularly of the amino acids, is considered insufficient to account for the relatively large amts. of *d*-lactic acid produced, especially since the non-protein N continues to increase at the time when *d*-lactic acid is decreasing. The conclusion is made that the amino acids present and formed during incubation are not transformed into *d*-lactic acid.

Period of Incubation.	Total non-protein, N		Non-protein N pptd. by phosphotungstic acid.		Amino-acid, N	
	White %.	Yolk %.	White %.	Yolk %.	White %.	Yolk %.
0.....	0.0032	0.0174	0.0013	0.0043	0.0002	0.0042
1.....	0.0029	0.0274	0.0012	0.0084	0.0002	0.0080
3.....	0.0073	0.0430	0.0036	0.0156	0.0007	0.0107
7.....	0.0141	0.0463	0.0077	0.0186	0.0013	0.0120
14.....	0.0214	0.0672	0.0088	0.0360	0.0019	0.0136

F. S. HAMMETT

The influence of the addition of glucose and alanine to egg-white on the formation of *d*-lactic acid during incubation. MASAJI TOMIYA. *Biochem. Z.* 116, 15-21(1921).—Measured amts. of glucose or alanine were aseptically injected into hen eggs through a small hole bored in one end, which was subsequently closed with paraffinized paper, and the eggs were incubated at 39.5°. After 3 days incubation those eggs which were proved to have developing embryos within were removed and the amt. of *d*-lactic acid formed in white and yolk was detd. and compared with the amts. found in untreated incubated eggs. The results showed an increase in *d*-lactic acid content of the whites of those eggs which has had the glucose injection. The yolk did not show this effect nor did alanine in either white or yolk produce any significant differences. Consequently M. believes that the formation of *d*-lactic acid from amino acids in the hen egg is doubtful, and that glucose is a participant in the process of the acid production.

F. S. HAMMETT

12—FOODS

W. D. BIGELOW AND A. E. STEVENSON

Report on coloring matters in foods. W. E. MATHESON. *Bur. of Chem. J. Assoc. Official Agr. Chem.* 4, 452-5(1921).—Notes on coloring matters in foods are given with special emphasis on the anthocyanins and glucoside fruit colors after the work of Willstaetter and Schudel (*C. A.* 13, 578; cf. also *C. A.* 15, 903). H. A. LEPPER

Guides to analysis. VII. The interpretation of food analysis. R. E. DOOLITTLE. *Chem. Bull. (Chicago)* 8, 94-6(1921).—The interpretation of the analytical findings of most food products is involved and difficult and is based on a careful study of many analyses of products of known origin covering variation due to stage of maturity, climatic conditions, soil, season and other sectional factors. Food is analyzed chiefly for purity and quality. Physical tests (macroscopic and microscopical) and bacteriological exams. are often necessary to detect adulteration. H. A. LEPPER

Food value of margarin. J. S. ABBOTT. *Chem. Age (N. Y.)* 29, 93-4(1921).—A discussion of digestibility and energy value of the fats usually used in margarin manuf.

From the literature A. states that all margarins have appreciable quantities of fat-sol. vitamins and some are quite able to replace butter in satisfying nutritive requirements. The process of manuf. does not affect the vitamins. H. A. LERRICK

Flavoring extracts in Canada's new Food and Drugs Act. ANON. *Spice Mill* 44, 690-1(1921).—Excerpt of clauses appertaining to flavoring exts. C. W. TRIGG

Report on maple products (for 1919). J. F. SNELL. *J. Assoc. Official Agr. Chemists* 4, 428-43(1921); cf. C. A. 15, 903.—Moisture tests by the refractometer have given fair agreement, and with careful temp. control the deviations need not exceed 0.3% of water. Cotton wool was found to be superior to filter paper as a filtering medium used in the prepn. of the sample. Pb subacetate solns. prepd. from Horne's dry salt were a little more variable in Pb content than those made from litharge and Pb acetate, but they were more uniform in basicity. Those made from Horne's dry salt gave slightly higher av. Winton lead numbers with 6 out of 7 samples. Results obtained for the Canadian lead number by different analysts varied considerably, higher results generally being obtained with the more basic Pb solns. But the results are evidently also affected by some other unrecognized condition. The ash values obtained by different workers agreed very closely. Moistening of the ash after most of the C has been burnt off facilitates ashing and largely prevents mechanical losses. Methyl red is used for detg. alkalinity of the ash, instead of menthyl orange. Cowles' method for detg. the malic acid value is not satisfactory. It is possible that a better method may be worked out with the use of CaCl_2 instead of the acetate; BaBr_2 might also be considered (C. A. 4, 290). In detg. the conductivity value close agreement was obtained by using always solns. containing exactly 22 g. dry matter per 100 cc. On the basis of the results obtained the following recommendations are made: That slight changes be made in the methods for prepn. of sample, detn. of total ash, and of Winton lead number; that the refractometric detn. of moisture be given preference, and that the other methods be referred to the referee on saccharine products; that the Canadian lead number method, and the conductivity method as described be adopted as tentative methods. F. W. ZURBAN

The use of glycerol in food flavors. MELVIN DE GROOT. *Tea and Coffee Trade J.* 40, 502, 504(1921).—A brief non-technical discussion of glycerol manuf., its chem. and physical properties. Considerable data are given in regard to its solvent action toward various substances, and particularly those of interest to the ext. maker. Its acceptability from a bromatological standpoint is emphasized. C. W. TRIGG

The action of hydrogen peroxide on flour. LINDET. *Compt. rend. agr. France* 6, 843 (1920).—Wheat grains contain a sol. ferment which decomposes H_2O_2 . The catalase is found mostly in the peripheral parts of the grain. White flour is poor in catalase. Based on these observations a process has been developed in which the purity of the flour may be ascertained as well as the fineness of the milling. Details of the process are not described. F. M. SCHERRE

The skin that forms on heated milk when left to stand. WILHELM MÜLLER. *Mitt. Lebensm. Hyg.* 12, 100-3(1921).—Heating milk to boiling, allowing to stand, filtering skin on a sieve (0.8 mm. mesh) and comparing fat content of the milk before and after heating showed that standing up to 5 hrs. did not materially lower this constituent in the milk. Increasing the time of standing defats the milk like skimming, (20% removed in 7 hrs.). Skin formed on standing 7 hrs. showed H_2O 48.45, fat 41.43, protein 4.71, lactose 3.34 and ash 0.69%. The protein consisted of casein with no albumin. H. A. LERRICK

Estimation of dry solids-not-fat in separated milk. R. ERMANN. *Milchwirtschaft. Zeits.* 48, 17(1920).—The fat-free dry substance in milk according to Fleischmann's formula corresponds to $v = 2.665 [(s-1)/s] 0.2 f$. If in this formula the wt. of unit vol. is replaced by the value of the unit wt., i. e., $s = 1/v$; then $v = 2665 (1-v) 0.2 f$.

Now $(1-v)$ is the reduction in vol. with increase in amount of v . If v represents the percent by vol. $v = 2665.1 (100-v) 0.2f$.

H. F. ZOLLER

Limitations of the reductase test for determining the quality of milk. O. RAHN. *Milchwirtschaft. Zentr.* 49, 287-90 (1920).—While the methylene blue reduction test has a certain value and is better than no test at all, the results obtained by it show many abnormalities, and the information yielded by the test is in no way as reliable as that given by a chem. and bacteriol. examn.

J. S. C. I.

Composition and analysis of milk preserved with dichromate. G. HINARD. *Ann. fals.* 13, 463-74 (1920).—The addition of dichromate (0.1%) to milk retards the development of the bacteria present, but does not destroy them. For a short time the results obtained on the analysis of a dichromated milk are comparable with those yielded by the fresh milk, but decompn. soon commences and affects the constituents to varying and different extents, so that it is not possible to correct the results obtained in order to ascertain the compn. of the original milk. Further, the presence of the dichromate itself affects the detn. of the total solids to an extent which varies with different milks.

J. S. C. I.

Simplified molecular constant for detecting added water in milk. M. SIROT AND G. JORRÉ. *Ann. fals.* 11, 88-92 (1918).

E. H.

Simplified molecular constant in the analysis of curdled milk. FONSES-DIAZON. *Ann. fals.* 12, 202-203 (1919).—Coagulation and decompn. of a sample of milk does not interfere with the use of the simplified mol. const. for detecting the presence of added water (preceding abstract); the acidity of the milk gives a measure of the quantity of lactose which has been decomposed, and this quantity added to the amt. of lactose still present gives the total lactose. The Cl is detd. in the serum and the data thus obtained are sufficient for calcg. the value of the simplified mol. constant of the milk in question.

J. S. C. I.

The chemistry of tea. CHRISTIAN BOEHRINGER. Stuttgart. *Chem. Ztg.* 45, 29-30 (1921).—Large amts. of caffeine may be recovered from prunings of the tea plant, the sap squeezed from tea-leaves during rolling, and "tea-down," the fine hairs broken from the leaves during firing. The latter, which contains 2-4% caffeine, is produced to the extent of 1.5% by wt. of the tea manufd.

C. W. TRIGG

The chemistry of tea. I, II. Caffeine and related compounds. CHARLES W. TRIGG. *Tea Coffee Trade J.* 40, 459-61, 606-8 (1921).—Important constituents of tea are enumerated and briefly discussed. Caffeine is considered with regard to its occurrence, distribution in the tea plant, effect of manuf. upon its % and state of existence in tea. Caffeine detn. and content in various teas are reviewed. Caffeine-free tea is contemplated from the mfg. and physiological standpoints. Bibliography.

C. W. T.

The sweetening power of saccharin and of dulcin. G. BRUHNS. *Centr. Zuckerind.* 29, 725 (1921).—B. comments on the results obtained by Paul (C. A. 15, 1361) that dulcin increases the sweetening power of saccharin, that mixts. of the two in certain proportions show a max. sweetening power, and that these mixts. have a more agreeable flavor than saccharin alone. B. explains this on the basis of the greater sweetening power of each in dil. solns., and concludes that the latter quality is additive. It is surprising, however, that this can be detected by the taste nerves, and it must be concluded that saccharin and dulcin excite different nerves. If this is the case, then there is a possibility of making mixts. of high sweetening power containing cheap sugars. Dulcin may also be used in such mixts., but not saccharin, on account of the objectionable flavor of the latter.

F. W. ZERRAN

A chemical method for the detection in fruit of a prior frozen condition. WILLIAM M. DEHN AND M. C. TAYLOR. *J. Ind. Eng. Chem.* 12, 977-9 (1920).—The method is based on the fact that freezing brings about a very rapid transformation of sucrose to invert sugar, the ratios of invert sugar to sucrose before and after freezing of the sam-

ple being compared. Great deterioration of fruit out of effective cold storage will invalidate the chem. detection of a prior frozen condition. H. A. LARPER.

Present status of fruit and vegetable dehydration. WM. V. CRUICK. *Chem. Met. Eng.* 24, 781-5(1921).—Com. operations in present activity are described and advantages of dehydration over canning are cited. The heat efficiency of evaporation, area of heating system necessary to heat the air, the vol. and velocity of air required, effect of humidity, recirculation, critical temp., and types of dehydrators are discussed. Cf. C. A. 15, 1045. H. A. LARPER.

Vinegar manufacture in the United States. PAUL HASSACK. *Chem. Age* (N. Y.) 29, 105-9(1921).—The general com. process for mfg. vinegar from black strap, corn or grain, malt, and cider are described. H. A. LARPER.

Detection and quantitative determination of preservatives in foodstuffs. FÉLIX DAEELS. *J. pharm. Belg.* 3, 273-274(1921).—To detect benzoic and salicylic acids, D. proceeds as follows: (1) *In milk*.—To 50 cc. of the liquid, add 5 cc. of a 10% $K_4Fe(CN)_6$ soln. and 5 cc. of a 40% $ZnSO_4$ soln., shake vigorously and filter. To the clear filtrate, add about 1 g. of $KHSO_4$. Make certain of the acidity of the soln. by testing with litmus paper. Shake out with 30 to 40 cc. of ether, collect the ether soln. in an evap. dish and heat on a water bath just to dryness. Cool the residue and wash with 4 to 5 cc. of cold distd. H_2O . Place the soln. in a test-tube, cool by holding it under a stream of water and add drop by drop a very dil. soln. of $FeCl_3$. If benzoic acid is present, a rose-salmon colored ppt. will be obtained. If the acid is salicylic, a violet color will develop. (2) *In butter, margarine or lard*.—Melt 50 g. of the substance in 200 cc. of hot distd. H_2O contained in a flask sufficiently large to permit of frequent shaking. Heat on a water bath for about $1\frac{1}{2}$ hr., allow the fat to sep., siphon off the aq. liquid and proceed as under milk. (3) *In conserves and pastes*.—Triturate 15 to 20 g. of the material in a mortar with a small amt. of distd. H_2O completely to disintegrate the mass; then ext. with hot distd. H_2O and proceed as described under milk. (4) *Quant. detn.*—Wash the ppt. obtained by the addition of $K_4Fe(CN)_6$ with 2 or 3 portions (20 to 30 cc. each) of boiling H_2O , filter the washings and add them to the original filtrate. Acidify the filtrate, and ext. with ether as described under milk. Finally, remove the ether by evapn. on a water bath at a low temp. and weigh the residue. A. G. DUMEZ.

Absorption of salt by meat and fish from a freezing mixture. S. SCHMIDT-NIELSEN. *Tekn. Ukeblad* 67, 456(1920).—When meat and fish are immersed in NaCl soln. at ordinary temp. they absorb considerable quantities of the salt but when they are placed in a mixt. of ice and salt at -5 to -15° , or the liquid mixt. of the same at this temp., the absorption of salt is very small and is negligible at a depth of 2 mm. below the surface of the meat. The penetration of the salt is the greater the higher the temp. of the liquid above its f. p. J. S. C. I.

Frozen meats. T. M. GARCIA AND J. S. FUSTER. *Rev. hig. sanidad pecuarias Madrid* 10, 246(1920).—From the analysis of a sample of frozen lamb and ox and a peptin digestion of fresh and frozen lamb and ox meat the authors conclude that the treatment of meat subjected to freezing does not impair its quality and digestibility. R. B. DUGGER.

Determination of water in cereal and meat products. F. C. COOK. *Bur. of Chem. J. Assoc. Official Agr. Chem.* 4, 347-51(1921).—Lower results for moisture (loss of weight) with malted cereals high in starch and higher results with those high in sugar were obtained in a vacuum oven at 65° for 14 hrs. than in a vacuum desiccator (H_2SO_4) for 5 days. Meat and fish, practically carbohydrate-free, gave identical results, while meat exts. gave higher results in the desiccator. H. A. LARPER.

Constituents and nutritive values of kao-liang (*Andropogon sorghum*, Brot.). JINRO KINURA. *J. Chem. Soc., Japan* 42, 163-80(1921).—Kao-liang belongs to the grain family and is one of the common foods used in Manchuria and northern China. Chem.

analysis shows that 100 parts of H₂O-free grain contain 11.23 parts of crude protein, 2.29 crude fat, 83.86 sol. non-nitrogenous substance, 1.43 crude fiber, and 1.16 ash. Its protein content is not less than that of rice, prolamine being the main constituent of the protein. It contains 5.02% arginine, 2.39% histidine and 2.96% lysine. From the alc.-sol. fraction of the protein, K. isolated 4.26% arginine, 3.67% histidine and 4.53% lysine. The total diamino acids, therefore, are less than those of rice, being about the same as those of wheat. Nutritive value was investigated by expts. with albino rats. It is not as great as that of rice, probably owing to lower content of diamino acids than in rice. If tryptophan is added together with diamino acids, the nutritive value of kao-liang protein becomes far greater. S. T.

Harvesting, storage and drying of barley. A. CLUSS, W. KLUGER and V. KOUDELKA. *Z. ges. Brauw.* 43, 353-8, 361-7(1920).—Different portions of the same crop of Original Hannchen barley were, after harvest, treated differently in respect of time of threshing, storage, and drying, and the effects of the various treatments on the grain were investigated. It is concluded that artificial drying of the grain is a valuable means of improving its quality for agricultural and brewing purposes; in particular the germinating power of the grain is markedly increased. The barley should be stored, preferably in the ear, for about 6 weeks before being dried, to permit after-ripening. Better results were obtained by reducing the moisture content from 17.9% to 11.8% than by reducing it only to 13.8%. After storage for about 2 months (in the air) the variously treated lots of grain attained practically the same moisture content, and underwent no further perceptible change in composition. J. S. C. I.

Commercial feeding stuffs. A. MCGILL. Dept. of Health, Ottawa. *J. Assoc. Official Agr. Chem.* 4, 351-60(1921).—The Commercial Feeding Stuffs Act of Canada (1909) and standards for bran, chop feeds, shorts, middlings and screenings are discussed. The presence of poisonous seeds in screenings is condemned. H. A. LEPPER

Report on stock feed adulteration. B. H. SILBERBERG. Bur. of Chem. *J. Assoc. Official Agr. Chem.* 4, 340-4(1921).—The method of S. studied in 1917 for hulls in cottonseed meal (C. A. 15, 714) was found to give varying results in the hands of various collaborators and further work on this detn. is proposed. Results by collaborators on a horse-feed containing corn, oats, barley, alfalfa, cottonseed meal, and molasses were unsatisfactory. Feed analysis must be done by a chemist and microscopist working together to obtain efficient results. H. A. LEPPER

Investigations of "sugar fodders" (and molasses mixtures) with respect to their sugar content. L. V. WISSELL. *J. Landw.* 65, 201-18(1917).—Unrefined sugar was mixed with oat straw meal in various proportions, with varying contents of water. Little or no change ensued on storage of the dry mixed materials, but the amt. of inversion in the mixts. which contained moisture became greater the longer the goods were stored. In the moist material (4% water) a musty smell developed and fungi became abundant. A moisture content of 1.3% showed no inversion while 2.2% showed slight inversion after 2.5 months. CaCO₃ added to a mixt. which contained 2.1% moisture showed some inversion. Sugar and peat meal with a moisture of 4.8% showed much inversion. Inversion takes place to a slight extent in 4 months with pure sugar with a moisture content of 2.6%. A mixt. of molasses and straw with a water content of 18.2% showed an increase of 3.1% invert sugar in 4 months, while with 30.4% of water the increase was 11.6%. Expts. were also made without disinfectants, with HgCl₂, and with thymol, while other samples were sterilized; the last was most effective in preventing change. Gravimetric methods of detg. sugars were considered more reliable than polarimetric methods. F. M. SCHERTZ

Indigenous stock feed versus imported materials. C. A. B. *Intern. Sugar J.* 23, 202-3(1921).—This is a report of the committee on stock feed of the Hawaiian Sugar Planters' Assn. Attention is called to the fact that as good or better rations may be

compounded from native materials than by using those brought from the main land. Cane tops, molasses, cassava, alfalfa, keawe bean meal, and a number of native plants and products made therefrom may be used successfully. The results from some exptl. mixts. are given.

F. W. ZERNAN

Enrichment of straw fodder with the protein of molds. H. PRINGSHEIM AND S. LICHTENSTEIN. *Cellulosechemie* 1, 29-39(1920); cf. C. A. 14, 580.—In expts. with a non-pathogenic *Aspergillus* species it was found possible to use NH_4Cl and a mixt. of kainite and superphosphate instead of $(\text{NH}_4)_2\text{SO}_4$ and K phosphate in the nutrient soln. The soln. is poured over the straw only once at the commencement, and subsequently daily turning of the couch is unnecessary. The depth of the couch should not exceed about 30 cm., in order to avoid undue rise of temp., which should be kept below 40° . Aeration of the resting couch with moist air appears very beneficial. Products contg. 8% of protein can be obtained.

J. S. C. I.

Detection of castor-bean meal in oil press cakes destined for animal feeding. C. BRIOUX AND M. GUERBERT. *Repert. pharm.* 33, 46-7(1921); cf. C. A. 14, 2225, 3484. B. and G. have studied two methods for the identification of castor oil press-cake in peanut and other oil press-cakes: (1) Microscopical, when the seeds have not been decorticated previous to pressing. (2) Agglutinin reaction, applied when the seeds have been decorticated. In the application of the former a 2% aq. soln. of KOH is added to the finely powdered meal and the whole brought to boiling. Upon decanting the liquid the castor bean hull remains undissolved if the meal is contaminated with castor bean. It has been shown that castor oil "ricine" in physiol. salt soln. agglutinates the blood of mammals and birds, even when present in traces. Therefore, in applying the reaction it is necessary to concentrate the albuminoids in the presscake by pptg. them with $(\text{NH}_4)_2\text{SO}_4$. The agglutinin reaction is applicable only to the identification of castor-bean meal in peanut press-cake. With corn meal, cotton seed, copra, rape seed, and poppy seed the corpuscles are slowly hemolized and develop a brown color. Linseed press-cake yields a mucilaginous mass which filters badly. Soy-bean cake contains an agglutinin similar to that of the castor bean.

H. F. ZOLLER

Suggested modification of the method for crude fiber. L. D. HAIGH. Univ. of Missouri. *J. Assoc. Official Agr. Chem.* 4, 338-9(1921).—It is suggested that feeds high in protein content, such as linseed and cottonseed meal, be digested at 40° for 30 min. in presence of dil. HCl and filtered on asbestos before the H_2SO_4 treatment of the official crude fiber method. No colloidal solns. are then encountered in the filtration to cause difficulty. Collaborative results did not warrant recommendation for adoption of the modification but further study is proposed.

H. A. LEFFER

Determination of the total sulfurous acid in organic substances (FROBOSER) 7. Detection of lactic acid (HARTWIG, SAAR) 7.

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THOMAS, KARL: Nahrung und Ernährung. Leipzig und Berlin: B. G. Teubner. M. 5 + 120% T.-Z. For review see *Z. angew. Chem.* 34, Aufsatzteil 63(1921).

Bleaching and conditioning flour and meal. J. C. BAKER. *Brit.* 159,166, Dec. 13,

1920. Flour and other cereal products are bleached and matured by treatment with NCl_3 , which is applied in the form of vapor mixed with air or other inert gas. Cl as a gas or in soln. is passed into liquid or mixed with a soln. of an NH_4 salt, and air is passed through the liquid to carry off the NCl_3 . The NH_4Cl may be treated with bleaching powder or NH_3 mixed with air or CO_2 may be passed over bleaching powder. Alternatively a soln. of Cl may be neutralized with limestone and mixed continuously with $(\text{NH}_4)_2\text{SO}_4$ soln. The liquid may be sprayed or aerated to vaporize the NCl_3 . Cf. *C. A.* 15, 1047.

Baking-powders; self-raising flours. C. S. BROCK. Brit. 157,581, Oct. 31, 1919. Powdered buttermilk is employed in part substitution for the usual acid component of baking powder or self-raising flour. Preferably 4.6 parts by wt. of powdered buttermilk, 2.1 parts of cream of tartar or the like and 1.3 parts of NaHCO_3 or its equiv. are added to 92 parts of flour. The baking powder may be dild. with corn-flour or rice flour.

Aromatizing edible oil and fat. JOHN DE BRUYN. Norw. 30,855, June 28, 1920. The pressed residue after the removal of fat from vegetable material is treated with weakly alk. water for 30 min. at $54-90^\circ$, the ext. then inoculated at about 21° with lactic acid bacteria to the end of acidulating it, without first adding sugar, salts or fat. The acidulated ext. is emulsified at $20-30^\circ$ with edible oil or edible fat in such a manner that the mixt. will contain 10-30% of acidulated ext.

13—GENERAL INDUSTRIAL CHEMISTRY

HARLAN S. MINER

[The place of chemistry in business.] A. D. LITTLE. *J. Ind. Eng. Chem.* 13, 386-90 (1921).—In this address before the Rochester Chamber of Commerce the story of the part which chemistry has played, is playing and can play in industry and commerce is well told in a manner to interest the business man. E. J. C.

The American chemical industry and its need for encouragement and protection. NICHOLAS LONGWORTH. *J. Ind. Eng. Chem.* 13, 384-6 (1921).—An address. E. J. C.

Post-graduate training in industrial chemistry. FRANCIS H. CARR. *J. Soc. Chem. Ind.* 40, 161-4R (1921). E. H.

The School of Chemical Engineering Practice of the Massachusetts Institute of Technology. R. T. HASLAM. *J. Ind. Eng. Chem.* 13, 465. E. J. C.

The case for chemical warfare. WILLIAM J. POPE. *Chem. Age (London)* 4, 526-8 (1921). E. H.

Some problems of national defense. JAMES W. WADSWORTH, JR. *J. Ind. Eng. Chem.* 13, 382-4 (1921).—An address. E. J. C.

Inorganic chemical industry in Germany during the war. GINO GALLI. *Giorn. chim. ind. applicata* 2, 511-2 (1920).—Review. ROBERT S. POSMONTIER

Breadth and scope of chemical patent claims. C. H. BIESTERFELD. *Chem. Met. Eng.* 24, 881-4 (1921). E. H.

Materials of construction used in a chemical works. GEORGE B. JONES. *Chem. Age (London)* 4, 394-6, 416-7 (1921).—All engineering materials used in chemical works must be viewed from 3 main aspects: (1) Physical properties, (2) economics, (3) chemical properties. The resistance of steel, cast iron, hard and soft lead, bronzes, high-silicon irons, certain special alloys, Zn, Al, Cu, Sn, Ni and Sb to corrosion is discussed, especially when subjected to the action of H_2SO_4 , HCl and HNO_3 under various conditions of concn. and temp. The design, limitations, etc., of castings made of high-silicon irons are gone into. The field for fused silica ware, glass, chemical stoneware, bricks, tiles and natural stone is indicated. Jointing materials and material for linings

such as vulcanite, asbestos mixts., sulfur mixts. and tar mixts. are taken up. Views on selecting building materials with special regard to cost, permanence, alteration, reconstruction and corrosion are expressed.

E. G. ROADMAN

The covering of chemical apparatus with metals and acid-resisting materials. F. SCHULER. *Chem. Ztg.* 45, 315-6(1921).—The selection of material for construction of equipment should be made with regard to inactivity toward materials to be handled, durability, ability to withstand necessary mechanical strains, and cost. Use of Al in place of Cu considerably decreases costs but at present chem. equipment is constructed of Fe and this covered with material suitable for the process requirements. Galvanizing is suitable for handling weak solns. and providing cleanliness in food processes. Pb and Sn coatings are not suitable for app. to be subjected to mechanical stresses. Autoclaves are constructed of Fe and covered with Pb. Enamels are not satisfactory where agitation, active chemicals, and changing temps. are encountered. Enameling offers low installation cost but the tendency to crack and chip are objectionable. Replacement is difficult and expensive. None of the above is suitable for handling active chemicals. For these stoneware, porcelain, or glass must be used either alone or as protection for Fe or Cu construction. Stoneware, successful in simple equipment, is so thick that the vol. of the app. is diminished and arched covers (tops) and agitators must be protected by alloys which cling to better advantage. The use of glass is especially recommended. The glass so used is thoroughly resistant; heating can be accomplished externally and heat losses are somewhat lower than those caused by Pb coverings. The glass is not easily injured by mechanical strains and no more care is needed in handling during transportation than for metal equipment. Cold liquids can be poured into heated glass-covered equipment with no danger of damage. The use of glass-lined Fe autoclaves and extractors is urged to replace expensive Cu construction.

G. R. JACKSON

Utilization of waste heat from technical furnaces and from chemical reactions. ERNST BLAU. *Chem. Ztg.* 45, 381-3(1921).—Under present economic conditions in Europe plants for utilizing waste heat can often pay for themselves in one year. Typical installations are described and illustrated. The claim is made that 10-18% of the heat energy of the fuel is thus saved.

W. C. KAUVEN

Drying theory; most economical temperatures; graphic determination of weight of moist air. R. HOEN. *Z. Ver. deut. Ing.* 63, 821-8(1919); *Mech. Eng.* 42, 160-71(1920); *Science Abstracts* 23B, 561-2.—A discussion of the most economical temp. and most economical degree of satn. for the air at the outlet from the drying chambers; discussion of influence of air pressure; and graphical methods for the solution of problems in connection with processes of drying. *Most economical temps.*—The fact that the highest possible temp. of air is the most economical in drying is well known. This is due to the fact that the ability of air to hold moisture rises very much more rapidly than does its temp. A good deal less information is available, however, as to the temp. at which the air should leave the drying chamber. As regards this latter, the impression seems to prevail that it is of advantage to have such air leave the drying chamber at the lowest possible temp. and the highest possible degree of satn. The assumption as regards the necessity of employing a high degree of satn. in order to ensure the economic operation of the drying process is correct, but the assumption as to the necessity of keeping the air at a low temp. rests on an erroneous conception of the character of the process. In order to clear up this matter, H. investigates how many g. of water each heat unit (kg.-cal.) contained in the air (considered as a mixt. of air and water vapor) can carry away. The wt. of 1 m³. of moist air is composed of ϕG_d kg. of water and $0.465 \frac{p - \phi p_s}{273 + t}$ kg. of dry air. In this formula ϕ is the degree of satn. in percent; G_d the wt. in kg. of 1 m³. of satd. steam at the temp. t ;

p the barometric pressure of the air-water vapor mixt. in mm. of Hg; p_d the steam pressure at the temp. t (really the partial pressure of the steam content in the mixt.) in mm. of Hg. Further, p_1 is the partial pressure of the air equal to $p - \phi p_d$ in mm. of Hg. In this formula G_d and p_d for a given temp. can be found in steam tables; the degree of satn. ϕ can be measured by means of a hygrometer, or more precisely by a psychrometer. The heat content of 1 m³. of mixt. at temp. t (or 1 m³. of the mixt. of the weight given above) is equal to $J = \phi G_d i + 0.465 \frac{p - \phi p_d}{273 + t} 0.24 i$ where i is the heat content of 1 kg. of steam at temp. t ; the coeff. 0.24 is the sp. ht. c_p of 1 kg. of air at const. pressure (all values in metric units). It is clear from the above that the wt. of water vapor corresponding to one heat-unit (kg.-cal.) content as part of the total heat content in 1 m³. of moist air is $\phi G_d / J$. It is desirable to plot this fraction as a function of temp. t in order to see what would be its max. and minimum values. To do this H. instead of resorting to the complicated analytical treatment of the problem simply computes the values of the numerator and denominator of the fraction and plots the quotients as coördinates with the temps. as abscissas. The conditions most easily attainable mechanically and still satisfactory from an economic point of view would lie, so far as temp. is concerned, between 60° and 80° and 60 to 80% of satn. in this case we expend roughly 1 kg.-cal. of heat to take care of 1.4 g. of water, which means an expenditure of 0.7 kg.-cal. of heat for 1 g. of water, or 700 kg.-cals. of heat for 1 kg. of water. This is very nearly the same as the heat necessary to evap. 1 kg. of water at atm. pressure, which is 640 kg.-cals. Under this condition the efficiency of drying is close to 90%.
H. G.

Calculation of percentage removal of a constituent from a mixture. W. B. VAN ARSDALE. *Chem. Met. Eng.* 24, 848(1921).—A triangular diagram is given with a network of straight lines reading "original percentage," "final percentage" and "percent removal" of a constituent. For gases the original and final percentages may be expressed by volume or by weight, the result being percent removal by weight in either case.
W. B. V.

Supplementary explanations of cold steam distillation. HEINRICH OFFERMAN. Peine. *Chem. Ztg.* 45, 322(1921); cf. *C. A.* 15, 309.—The neck of the retort should be elevated just enough to cause particles spit over to run back. It is not necessary to depend on thermometer temps. when starting in the steam; the steam-generating flask should be connected to the retort at the moment when the contents of the latter are at a full boil, with a little coming over, and just as the H₂O begins to boil; otherwise foaming or frothing may occur. If the distillate solidifies readily it is well to insert an air-cooled flask before the H₂O cooler, in some cases keeping the flask at 105–10° in order to drive the H₂O and lighter essences on to the H₂O cooler.
J. H. MOORE

Evaporation from reservoirs. H. A. BARRE, et al. *J. Elec. Western Ind.* 46, 489–96 (1921).—A compilation of empirical data recently obtained by a number of engineers, of particular interest to the chem. engineer confronted with the problem of storing water in tanks or reservoirs. Detailed tables, curves and literature references are given.
C. G. F.

Lubricators and lubrication. L. ARCHBUTT. *Chem. Age* (London) 4, 280–1(1921); *Oil and Gas J.* 19, No. 45, 58(1921).—Report of a lecture describing expts. on the use of fatty acids in mineral oils to obtain the "oiliness" usually obtained by adding much larger quantities of fatty oils. With a load of 270 lbs. per sq. in. and a speed of 7 ft. per min. 2½% of an oil containing 25% free fatty acid lowered the friction as much as 10%.
EUGENE C. BINGHAM

Colloidal chemistry is useful in many industrial processes. ISMAR GINSBERG. *Oil, Paint, and Drug Rep.* 99, No. 22, 24(1921).—The "colloidal mill" has enabled

efficient recovery of petroleum oils from muds and shales, and vegetable oils from press cake; the production of colloidal fuel oils, and colloidal copal varnishes; has been found useful in phosphatic rock and rosin extrs.; and in accelerating hydrogenation and sapon. of oils, etc.

F. A. WATTS

Permeation of oxygen breathing apparatus by gases and vapors. A. C. FIELDMAN, S. H. KATZ AND S. P. KINNEY. *Bur. Mines, Tech. Paper 272*, 24(1921).—The rubber breathing bags of O app. in vapor of volatile casing-head gasoline showed dangerous penetration; in 1 test 2.6% of gasoline vapor was in the bag after 15 min. exposure to air contg. about 34% gasoline vapor. All the fabrics now used by the Bur. of Mines for breathing bags, excepting the Fleuss, proved permeable to gasoline and C_2H_4 vapors (time of test, 2 hrs.) and undoubtedly to other similar org. vapors. No permeation of the fabric investigated was found for CO or natural gas. The impermeable Fleuss material consisted of heavy sheet rubber, $1/16$ in. thick, made of high-quality stocks; no cloth was used in it. Fabrics made of 2 rubberized sheets cemented with a glue and glycerol mixt. were found completely impermeable. One such fabric had a total thickness $1/4$ that required for rubber. Special tests for resistance to rough mechanical treatment, exposure to weather, hot dry air and freezing temp. were passed by the fabric without permeation or loss of flexibility. A fabric made of cloth impregnated and coated on one side with pyroxylin varnish allowed only a slight penetration of gasoline and C_2H_4 vapors towards the close of the 2-hr. test period, not enough to be dangerous. A slightly thicker coating would entirely prevent penetration. This fabric has apparently very desirable properties for use in breathing bags and deteriorates less than rubber with age.

C. J. WEST

The vapor compression system of evaporation (CARLSSON) 1.

DANCKWORT, PAUL W.: *Sibirien und seine wirtschaftliche Zukunft*. Leipzig and Berlin: B. G. Teubner. M. 12, bound M. 15. For review see *Z. angew. Chem.* 1921, *Wirtschaftlicher Teil*, 119.

Denkschrift der Aussenhandelsstelle Chemie. BERLIN, W.: Geschäftsstelle der Aussenhandelsstelle Chemie, Matthäikirchstrasse 9, M. 1.20. For review see *Z. angew. Chem.* 1921, *Wirtschaftlicher Teil*, 98.

GROSSMAN, J.: *The elements of chemical engineering*. 2nd Ed. revized. Preface by Sir William Ramsay. London: Ch. Griffin & Co. For review see *Giorn. chim. ind. applicata*. 3, 182(1921).

Historical review of the object organization and activities of the Chemical Alliance, Inc., during the world war, 1917-1918. New York: The Chemical Alliance. 83 pp.

Evaporation of solutions. GEORGE ARTHUR BUHL. *Norw.* 30,636, Apr. 19, 1920. The boiling down is effected with the aid of the principle known in the art of drying, according to which circulating superheated vapor of the liquid under treatment is used as only drying agent, the soln. to be evapd. being atomized into circulating steam. The surplus vapor escapes as such from the system, while the solid materials which sep. are collected in the circulation system.

Process and apparatus for degasifying and deaerating liquids, more particularly water. RASMUSSEN & ERNST, G.M.B.H. *Ger.* 322,887, Feb. 17, 1916. The liquid flows into a closed tank fitted with a deaerating tube, which during operation must at all times be completely filled with the liquid, in the direction of the air-discharge point, and at such a distance from the latter and from the bottom of the tank as to enable a sepn. into liquid relatively rich in air and hence of lower sp. gr., and liquid relatively poor in air and hence of higher sp. gr., the latter liquid being continuously drawn off from the

bottom. The upper part of the tank is fitted with cells or other devices subdividing the liquid current.

Drying colloids. G. P. LUNT. Brit. 159,465, Feb. 25, 1920; cf. C. A. 14, 2059. In recovery of solvent from nitrocellulose explosives or other colloid substances such as rubber the amt. and proportions of the different constituents of the solvent condensed are regulated by regulating the temp. and vol. of the condensing liquids, so that evapn. takes place at a rate equal to that at which the solvent diffuses through the colloid, thus avoiding "histering." The colloid is placed on sliding shelves in a chamber heated by hot-water pipes. Dampers are provided for cutting the chamber out of the circuit while it is being recharged, and an outlet is provided with a damper, whereby air can be displaced when a heavy gas, such as CO_2 or SO_2 , is used in the circuit. The gas is circulated by a pump and passes through a condenser, provided with jets of the condensing liquid, and a baffle for catching the spray.

Distilling oils, etc. W. J. MELLERSH-JACKSON. Brit. 159,774, April 14, 1920. In distg. oil or other liquid, oil from a tank is forced by a pump through horizontal pipes connected in series and heated by steam pipes passing through them. The lower pipes receive steam directly from a boiler and the exhaust steam is used to preheat the oil in the tank. The upper pipes receive steam from a superheater. The heated oil from the upper end of the coil passes through valves which retain a pressure in the coil and is sprayed into a drum from which the vapors pass to a condenser. In distg. oil, a temp. of $230\text{--}300^\circ\text{F.}$ is employed in the pipes and a temp. of $500\text{--}600^\circ\text{F.}$ in the pipes, and cracking of oil takes place. The cracking is facilitated by treating the oil in the tank with a reagent such as HCHO and H_2SO_4 .

Process of and apparatus for treating materials in roasting and drying. O. SPINZIG. Can. 210,249, Apr. 12, 1921.

Electric conductors. BRITISH INSULATED & HELSBY CABLES, LTD. and E. A. BAYLES. Brit. 159,760, March 2, 1920. Flexible or other single or double conductors are coated with an extra rubber sheathing capable of being dyed after vulcanization. The rubber insulation may also be made in one with the sheathing to give the required thickness. The vulcanized conductor is passed through a bath of dye of the desired color. The dyes employed are such as are sol. in CHCl_3 , benzene, naphtha, etc. In some cases H_2O solns. may be used. The colored strands may be made up into cables.

Insulating and protecting compositions. BRITISH THOMSON-HOUSTON CO., LTD. Brit. 159,421, March 17, 1920. A compn. for insulating and protecting electrical app., applicable for use as a cement, *e. g.*, for sealing electrode wires in spark-plugs, etc., or for coating resistance coils, etc., by dipping or otherwise, consists of silicate of Na or other alkali metal, mixed with a soln. in aq. NH_4OH of a resin or natural gum such as shellac, copal or colophony. A coloring matter such as ultramarine blue and a filler such as mica, feldspar, whiting or chalk may be added. Cf. 6927, 1887 and 18,953, 1898.

Coating surfaces. R. LAMP'N. (N&E MULLER). Brit. 157,812, Jan. 10, 1921. Cylindrical surfaces such as those of printing rollers are coated with solns. by rotating them in front of a reciprocating spray; or the spray may be fixed and the surfaces be reciprocated as well as rotated. The process will produce granulated as well as continuous coatings.

14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

Report on water. J. W. SALE. *J. Assoc. Official Agr. Chem.* 4, 380-8(1921); cf. C. A. 15, 910.—S. gives analytical data and suggests methods for the detn of I and Br in mineral waters and brines and the ammonias in waters containing sulfides. He recommends (1) that the detn. of Ba by weighing as BaCrO_4 or titration with iodide

and thiosulfate be adopted as official; (2) that the Na bismuthate method for the detn. of Mn be adopted as official; (3) that the detn. of I in the presence of Cl and Br by oxidation with KMnO_4 and titration with iodide and thiosulfate be adopted as a tentative method; (4) that the detn. of Br in the presence of Cl but not I with Cr_2O_7 and H_2O_2 and titration with iodide and thiosulfate be adopted as a tentative method; (5) that the detn. of the ammonias after boiling free from sulfide upon acidifying with H_2SO_4 be adopted as official; (6) that the methods on water be extended to cover the examn. of allied products, such as brine and salt; (7) that continued study be given to the detn. of I and Br and the heavy metals and to the use of equivs. in studying the character of waters.

G. C. BAKER

Composition of the Harrogate mineral waters. WM. LOWSON. *Analyst* 46, 125-8 (1921).—Analyses of the more important wells are given. They may be divided into 2 main groups, viz. sulfur waters and iron waters.

G. C. BAKER

Detection of very small quantities of indican in water as an aid to hygienic water analysis. A. JOLLES. *Ber. pharm. Ges.* 30, 421-2 (1920); *J. Chem. Soc.* 120, II, 69 (1921).—The presence of the merest traces of indican in a water is a certain indication of previous contamination with animal excreta. As its detection by chem. means is very simple and not interfered with by the commonly occurring constituents of natural waters excepting nitrites, which are readily eliminated by means of ferrous salts, its presence or absence forms an excellent criterion for the judgment of a water from the hygienic point of view. The test is as follows: Evap. 3-4 l. of the water to 250 cc., and if nitrites are present add 3 g. $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ for every 0.1 g. of nitrite per l. of the original water. Continue evapn. to 10 cc. and add to the filtered soln. 1 cc. 5 % alc. thymol or α -naphthol soln. and 10 cc. fuming HCl containing 5 g. FeCl_3 per l. After keeping for 15 min. with frequent shaking, ext. by careful shaking with 4 cc. of CHCl_3 . The indolignone coloring matter will have been formed if indican were originally present. A reddish or bluish violet color of the CHCl_3 layer, however slight, is a certain proof of the presence of indican, and of the previous contamination of the H_2O with animal excreta.

W. O. E.

Practical means of preventing corrosion of iron and steel where not exposed directly to the atmosphere. F. N. SPELLER. *Trans. Am. Electrochem. Soc.* 39 (Preprint); *Chem. Met. Eng.* 24, 1009-12 (1921); cf. *C. A.* 14, 2610.—Elimination of O is effected by de-aerating the water mechanically (this involves passing the water over baffles or spraying into a high-vacuum chamber at normal temp.) or by increasing the temp. and controlling the pressure and temp. so that more or less complete removal of all the gases is obtained. Almost perfect removal of gases is accomplished by heating the water above the b. p. corresponding to the pressure and spraying into a chamber at lower pressure, and having the air and vapor drawn through a condenser where the vapor is condensed and the heat transferred to the incoming water. To prevent internal corrosion of water pipes the O need not be lower than 0.4 cc. per l. but to protect steam boilers, superheaters and economizers operating at higher temps. the O must be below 0.2 cc. per l. A deaerator to accomplish this without heating the water to the b. p. is illustrated. A second means of protection is based on fixing the O by chem. combination. O is removed by combination with scrap steel sheets in a deactivator. The heated water is passed through a storage tank carrying expanded steel sheets. A combination of the mechanical deaeration and O fixation methods makes the water practically inactive toward Fe. One table shows small corrosive action of deactivated as compared with untreated water on Zn plates, steel and brass plates, steel and Cu plates and brass plates; another table gives a summary of research work of the past 5 years on the corrosion of pipes in hot water feed service. (See also *Chem. Met. Eng.* 24, 841-44 (1921)).

W. H. BOYNTON

The occurrence of hydrogen sulfide in deep layers of water. O. L'ONG. Brunswick.

Z. Nahr. Genussm. 41, 29-31(1921).—Water pumped from the bottom of a reservoir in the Oker river in the city of Brunswick was rich in H_2S , although the water running into the reservoir was free from H_2S . Just after the discovery of this condition the reservoir was emptied and further investigation was thus prevented. D. B. DILL

Bacteriological diagnosis for the pollution of drinking water. Contribution to the study of the reactions of culture media to neutral red. M. BORNAND. *Mitt. Lebensm. Hyg.* 12, 92-100(1921).—The addition of sugar agar to the neutral red media is recommended. On planting, a polluted water gives a canary-yellow fluorescence, eventually accompanied by gas formation. Sugar agar may also be added to congo red media.

G. C. BAKER

Water purification plant at Highland Park, Michigan. HOAD & DECKER. *Eng. News-Record* 86, 772-5(1921).—The plant consists of a 48,000,000 gal. concrete storage reservoir; a 2,750,000 gal. covered coagulation basin; a 12,000,000 gal. daily filtration plant and pumping station. The supply is obtained from Lake St. Clair, 11 miles distant. The water is pumped to a storage reservoir giving 3 to 6 days storage before passing to the coagulating basin. Details of design are given.

FRANK BACHMANN

Kewanee water works. ANON. *Power Plant Eng.* 25, 497-500(1921).—The Kewanee, Ill., plant comprises 2 distinct systems, one the air system to bring the water to the surface and the other the pumping system which serves the city mains. The changes consisted in sinking a new and deeper well, in building a new reservoir, in replacing the air compressor by a larger unit, and in putting in additional pumping and boiler capacity. The water employed for boiler feed is softened with lime-soda.

G. C. BAKER

Why Chicago does not filter its water. ANON. *Eng. News-Record* 86, 707(1921).—The principal reason is the high daily consumption of 265 gal. per capita. Until this rate is at least halved by the installation of meters, filtration advocates have little hope. By the use of Cl for disinfection of the water supply, the typhoid rate in 1920 was only 1.1 per 100,000, the lowest of any of the large cities. Objectionable tastes due to industrial wastes discharged into Lake Michigan have been the cause of many complaints.

FRANK BACHMANN

Comparison of three filters near Chicago. S. A. GREELEY. *Eng. News-Record* 86, 730(1921).—Operations of Evanston, Ill., Whiting and East Chicago, Ind., filters are compared. The Evanston plant has only pollution of domestic origin to deal with while the Indiana plants are handicapped by industrial pollution of the water supply. Relative detention periods of the 3 plants are $1\frac{1}{4}$ hrs. for Evanston; $3\frac{1}{4}$ hrs. for East Chicago and $4\frac{1}{4}$ hrs. for Whiting. The length of runs at Evanston vary from 7-8 hrs. to several days. Alum applied averages 0.7 gr. per gal. Wash water ranges from 1.3 to 1.6%. At East Chicago, the capacity of the plant was exceeded by 25%. Runs of 3.6 hrs. are obtained; wash water, 11.1%; and dosage of alum 1.2 gr. per gal. During times of rated capacity, runs of 6 hrs. and wash water of 6% were recorded. At Whiting, the runs averaged 9.4 hrs.; chemicals used were alum—1.5 and 1.4 gr. and lime, 0.7-0.8 g. per gal. Aerators are provided to remove oily tastes and odors from the latter supply. A distinct improvement is noted by the use of the aerators.

FRANK BACHMANN

Filter underdrains, sand bed and wash water experience. MALCOLM PIRNIE. *Eng. News-Record* 86, 371-2(1921).—Further discussion of symposium on underdrains, sand bed and wash water (see C. A. 15, 405).

FRANK BACHMANN

Watchman, sole attendant at filtered-water plant supplying people of Langeloth. DONALD J. BAKER. *Coal Age* 19, 750-4(1921).—The plant which serves a population of 2500, obtains its supply from an impounding reservoir covering eleven acres, and holding 3,000,000 gals. The water, although supplied by mountain streams and springs

is decolorized, clarified and sterilized before use and even that employed for fire fighting passes all the degrees of purification. The installation consists of a sedimentation basin $80 \times 20 \times 13\frac{1}{4}$ ft. deep, 2 filter units $10 \times 7\frac{1}{4} \times 8$ ft. deep and furnishes $\frac{1}{4}$ mil. gals. daily. All chemicals are automatically fed. G. C. BAKER

Zero water for boiler feed. LEROY W. ALLISON. *Power Plant Eng.* 25, 501-4(1921).—Zeolite softeners deliver water of zero hardness and boilers are maintained without scale while leaks and failures are instantly visible. No trouble is experienced from foaming. Such an installation will pay for itself in from 24 to 36 months. G. C. BAKER

The Riensch-Wurl sewage screening plant at Santa Barbara. ORMOND A. STONE. *Eng. News-Record* 86, 719-21(1921).—The screen is 10 ft. in diam. set on a 15° slope and has $\frac{1}{16}$ by 2-in. slots. Its max. capacity is 7 cu. ft. per sec. with a head of 18 in. Screenings are deposited by brushes into a pit from whence they are pumped by a special pump to an adjacent lot and composited with street sweepings. The solids from the screen average about 5 cu. yd. daily. FRANK BACHMANN

New ocean outfall and screens for Los Angeles sewage. W. T. KNOWLTON. *Eng. News-Record* 86, 331-2(1921).—Additional sewers are contemplated to take care of the fast growing city and suburban population of Los Angeles. Treatment of the sewage by fine screens of the drum type is considered sufficient for discharge into the ocean. Complaints on account of sewage solids washed on bathing beaches will cease after the sewage is screened. FRANK BACHMANN

Illinois sewage testing station. ARTHUR M. BUSWELL. *Eng. News-Record* 86, 378(1921).—The testing equipment includes a Dorrco screen, two combined aerating and settling tanks of the Dorr-Peck type, a sludge storage tank and a steam boiler. Sludge dewatering expts. are being made with an Oliver continuous filter and a Tolhurst centrifuge. The screen is 4 ft. in diam., 8 in. wide and revolves at 23 r. p. m. The screening medium is punched plate with $\frac{1}{16}$ by $\frac{1}{2}$ in. slots. The screen passes about 200,000 gals. sewage per day with a loss of head of 3 in. Screenings are scooped from the pit once a day by means of a perforated bucket. The Dorr-Peck activated sludge system consists of 2 round wooden tanks, each 17 ft. in diam. by 13 ft. deep. Trays are provided in the upper part of the tanks for sedimentation. When treating 100,000 gals. per day, the aeration period in the first tank was 3.2 hrs., settling 1.8 hrs., and in the second tank, aeration 2.8 hrs., settling 2.2 hrs., giving a total time of passage through both tanks of 10 hrs. with 6 hrs. aeration. Less than 1 cu. ft. of air per gal. is required to treat Champaign sewage, which is a strong domestic sewage. FRANK BACHMANN

Sewage disposal at Worcester, Mass. R. S. LANPHEAR. *J. Boston Soc. Civil Eng.* 8, 109-22(1921); *Public Works* 50, 421-3(1921); *Eng. Contr.* 55, 464(1921); cf. C. A. 14, 1865.—The developments of the Worcester plant are detailed. Chem. pptn. was begun in March, 1890. In 1900, settled sewage was applied to sand filters. Exptl. work was begun (1905) with contact beds. Later in 1911 Imhoff tanks and sprinkling filters were tried. A rate of filtration of 2 mil. gal. per 24 hr. was attained on stone $\frac{1}{4}$ to $2\frac{1}{8}$ in. and on $\frac{1}{2}$ to $1\frac{1}{2}$ in. In 1917 activated sludge was tried. In 1919 the adoption of the Imhoff tank and sprinkling filter was recommended. In the expts. on activated sludge, the sewage passed through a bar screen with $\frac{1}{4}$ in. openings, a grit chamber, entering the aeration tank (capacity 25,283 gal.) with filtros plates, passing to a settling tank of the Dortmund pattern. A sludge regeneration tank was provided (4386 gal. capacity). From 75,000 to 125,000 gal. per 24 hr. of sewage were treated, 2 to 5 cu. ft. air per gal. with an aeration period of 3.8 to 6.2 hours being used. The sludge was aerated 2.8 to 6.5 hours. Nitrification was absent, but thorough clarification was produced. To produce a stable effluent (75 to 90% stability) 4 cu. ft. air per gal. sewage, 6 hr. aeration of sewage, 6 hr. aeration of sludge, 25% returned sludge are recommended. From 12,000 to 25,000 gal. sludge per mil. gal. sewage resulted, with 97.1 to 98.7% moisture. Dried sludge contained 4 to 6.5% N with 8.5 to 18% Fe. Drying on sand beds

was impracticable. Concn. by settling did not reduce the H_2O content below 95%. Sludge pressing was possible with the use of 2,500 lb. lime to 100,000 gal. sludge.

LANGDON PEARSE

Water supply and other sanitation in Greece. WALTER E. SPEAR. *Eng. News-Record* 86, 708-15(1921).—An outline is given of the climate, meteorology and hydrology of Greece. Water supplies are scanty and sewers and other sanitary facilities are lacking. Much malaria exists owing to stagnant swampy areas near the sea and unprotected water supplies which serve as breeding grounds for mosquitos. F. B.

Fight against typhus in Poland. C. NASLUND. *Hygiea, Stockholm* 82, 845(1920); *J. Am. Med. Assoc.* 76, 830(1921).—Various measures were tried for clearing houses of lice. HCN was found effective beyond all anticipations. The concn. of the gas should be 0.5 to 2% depending upon the impermeability of the house. L. W. RIGGS

The improvement of oiling fluids for anti-mosquito work. W. NORMAN LEAK. *J. Trop. Med.* 24, 37-9(1921).—One % of castor oil increased the spreading power of paraffin oil 25 times and gives a tougher film. H. V. ATKINSON

The influence of the composition of water in its use for tanning purposes (GRASSER) 29.

HILGERMANN, ROBERT AND ZITKE, AUGUST: Grundlinien für die chemische Untersuchung von Wasser und Abwasser. 2nd Ed. revized. Jena: Gustav Fischer. M. 3.20, bound M. 4.20. For review see *Oesterr. Chem. Zig.* 24, 436(1921).

Purification of water for ice manufacture. HEINRICH WEHNER. *Ger.* 323,299, Nov. 15, 1918. The H_2O is treated with lime water and then deaerated *in vacuo*. Before the deaerating process a suitable acid is mixed with the H_2O .

15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

J. J. SKINNER

Chlorosis of lupines on calcareous soil. FRITZ MERKENSCHLAGER. *Fühling's Landw. Zig.* 70, 19-24(1921).—Chlorosis is not due to hunger but to poisoning. Physical properties of the soil do not cause chlorosis while thorough aeration of the soil reduces chlorosis. F. M. SCHERTZ

Report of the results obtained with reference to the natural nitrification of soils, based upon the study of drainage waters of 1906 and 1907. E. NORIEGA. *Rev. agr. (Colombia)* 3, 525-60(1919).—The expts. reported include 6 soil types of Colombia and were conducted in lysimeters holding from 36 to 40 kg. of soil. Total rainfall and drainage water recovered during the expts. is reported, with nitrate N of the latter. A rather large number of practical conclusions are given by N. but are not repeated because he considers them only tentative. The work is still in progress. R. B. DERMER

The movement of soil moisture. WILLARD GARDNER AND JOHN H. WIDSTON. *Soil Science* 11, 215-32(1921).—A general equation is developed for the movement of moisture through an ideal soil, based upon the assumptions that one, the inherent moisture cond. is independent of the moisture content; two, the capillarity potential is a linear function of the reciprocal of the moisture content. The application of the formula to several special cases is given. JEROME ALEXANDER

Estimation of the acidity of humic soils. S. ODÉN. *Int. Mitteil. Bodenkunde* 9, 361-74(1919); cf. *C. A.* 14, 3737.—An estn. of the total acidity of a soil does not show the amt. of humic acid, as a number of other acids are also present. The H-ion content of a soil is first detd. by the potentiometer method; concd. KCl is then added to the test portion of the soil, and the H-ion content is again measured. The acids in the filtrate are also estd. by titration, and the total acidity and acidity are detd. by the

method of Tacke or Tacke-Süchting (*C. A.* 3, 467), whereby an indication of percentage of acids present which readily form only slightly sol. Ca. salts is obtained.

J. S. C. I.

Studies on the decomposition of cellulose in manure and in the soil. CURT MÜTTERLING. Univ. Leipzig. *Diss.* 1913, 100 pp.—The results of the expts. show that contrary to the prevalent opinion that anaerobic bacteria are responsible for the decompn. of cellulose in nature, aerobic organisms, especially fungi and perhaps also actinomycetes, are of chief importance in this process. The dark discoloration of the paper which appeared in some cultures indicates the formation of humus substances. A high moisture content is essential for vigorous decompn. The qualifying influence of the culture medium was marked with some of the pure cultures. Temp. also had a very important influence, thus the appearance of decompn. was most frequently after 13 days at 16–22°, 7–11 days at 38° and 2–3 days at 50°. The decompn. of cellulose is not a restricted power of a small group of bacteria but many organisms possess it, particularly numerous fungi. The isolation of cellulose-destroying fungi is very easy while that of the cellulose-dissolving bacteria is extremely difficult. ALBERT R. MEES

Changes of potassium ammonium nitrate in the soil. NIKOLAUS KEMPF. *Landw. Versuchsst.* 97, 195–217(1921).— NH_4NO_3 , a war product, did not meet with favor by agricultural Germany, so the salt was mixed with other fertilizing salts as potassium. When the salt was tested on soils the univalent bases (NH_4 , K, and Na) were found to be the less sol. while the bivalent bases (Ca and Mg) were more sol. F. M. SCHERZ

Plant analysis and manuring. F. MÜNTER. *J. Landw.* 68, 207–224(1920); *J. Soc. Chem. Ind.* 39, 758A.—Barley and sugar beets were experimented on for 5 years and were grown in a loam soil fertilized with stable manure, nitrate, potash salts and phosphates separately and in combination. The Ca and Mg contents of barley were little affected but the Si of the straw was decreased by N and P applications to the soil. The N content was increased by K and P. Definite deductions were difficult to draw because of the weather factor. The roots of sugar beets were little affected by manuring, whereas Ca and Mg in the tops were increased by K manuring and decreased by P. The weather factor also greatly influenced the growth of beets so that deductions were difficult to draw regarding the needs of soils for particular fertilizers. For this last purpose 2 differently manured plots must be compared, one fertilized with N and the other with K and P. Under these conditions the following statements may be made: If on the N plot the ratio of N to K is greater than unity the soil is deficient in K; if on the same plot the ratio of N to P is greater than 100:20 the soil is deficient in P; while if on the P and K plot the latter ratio is less than 100:35, the soil is deficient in N.

F. M. SCHERZ

The cause of nitrogen losses from urine, feces and other organic substances. C. NOLTE AND E. POMMER. *Landw. Versuchsst.* 97, 245–260(1921).—A report of lab. expts. on losses of N from urine, etc. F. M. SCHERZ

The phosphates of Nauru and Ocean Islands. THOMAS STEEL. *J. Soc. Chem. Ind.* 40, 50–61T(1921).—Analyses of 13 phosphate rock samples from Nauru and Ocean Islands show a P_2O_5 content ranging from 31.76 to 40.44%. Only traces of Fe and Al with varying small amts. of F were found in any of the samples. W. H. ROSS

Need for lime as indicated by relative toxicity of acid soil conditions to different crops. BURT L. HARTWELL. *J. Am. Soc. Agron.* 13, 108–12(1921).—This paper emphasizes that the amt. of lime to be applied to a soil is detd. by the plant to be grown.

F. M. SCHERZ

The fertilizer problem. MARCHAIGER AND GOUJON. *J. pharm. chim.* 23, 171–80(1921).—The indiscriminate use of fertilizers is criticized. The one-sided prevalence of one constituent over another brings about profound changes in the organism of the plant, rendering it liable to attack by parasitic growths. The ideal fertilizer should

have a harmonious compn. that will stimulate, not hinder the antiparasitic action of the cell juice. The return of animal waste matters to the soil is essential. S. W.

Investigation of the action of fertilizers. LEMMERMANN. *Biedermann's Zentr.* 49, 291-310(1920); cf. C. A. 14, 995.—A continuous series of expts. with nitrogenous, phosphatic, potash, and lime fertilizers and stable manure showed that generally the fertilizers had at first but little effect on the crop yields, the exception being the effect of the nitrogenous fertilizers. J. S. C. I.

Composition of fertilizer prepared by treating lime with end-liquors from potash works. J. GÖRANSSON. *Z. öffentl. Chem.* 26, 205-13(1920).—Lime fertilizers, prep'd. by sprinkling burned lime with potash end-liquors contg. about 30% of $MgCl_2$, are of complex compn., the assumption that they are merely mixts. of $Ca(OH)_2$ and $MgCl_2$, with traces of $Mg(OH)_2$ and $CaCl_2$, being erroneous. A sample exam'd. consisted of a mass of damp, crumbly portions interspersed with hard lumps. An av. sample had the following compn.: Moisture (at 120°), 30.41; CaO , 27.49; MgO , 11.37; K_2O , 0.58; Na_2O , 0.42; Fe_2O_3 and Al_2O_3 , 1.18; SiO_2 (sol.), 1.24; SiO_2 (insol.), 0.66; Cl , 11.87; CO_2 , 4.08; SO_3 , 1.76%; P_2O_5 , Br. and org. impurities, traces; water of crystn. and OH combined with lime and $Mg(OH)_2$ (by difference), 7.83%. A sample taken from the hard lumps gave: Moisture (at 120°), 31.88; CaO , 36.63; MgO , 5.91; Cl , 4.89; CO_2 , 1.85%. This sample gave a dry, non-hygroscopic powder which implies presence of oxychlorides. The decompn. of these in the soil is dependent on conditions of time, temp., and concn. A lab. sample of the fertilizer treated with water on a filter gave a soln. contg.: CaO , 15.12; Cl , 11.75; K_2O , 0.60; Na_2O , 0.39; SiO_2 (sol.), 0.42; SO_3 , 1.39%; with traces only of MgO , thus confirming the absence of $MgCl_2$ from the original material. The residue contained CaO , 12.4; MgO , 11.4% and was of a light, pulverulent nature, and this condition was also produced with material exposed in the open for about six months, owing to atm. influences liberating the magnesia, which in the original material is present in a very finely divided condition and enclosed by $CaCl_2$. The liberated magnesia is in a very suitable form for distribution throughout the soil. Some photo-micrographs are appended. J. S. C. I.

Gas-volumetric determination of ammoniacal, nitric and organic nitrogen in mixed fertilizers. PRIMO BALDI. *Giorn. chim. ind. applicata* 2, 376-8(1920).—This study was applied particularly to ammoniacal-nitrated peats. The analysis comprized 3 detns. of NH_4 followed out by the hypobromite method. The first detn. is made upon the aq. soln. of the fertilizer, the 2nd detn. upon the aq. soln. after reduction of the nitrated N to NH_4 , and the 3rd upon the insol. residue after Kjeldahlizing the latter. B. employed the Davison-Parson reductor. Exhaust 10 g. of the fertilizer with warm H_2O , filter, and bring the filtrate to 200 cc. (soln. A). Det. the ammoniacal N on 10 cc. of this soln. in the usual manner by hypobromite. Take another 50 cc. of soln. A and treat with Devarda alloy (4-5 g.) and 100 cc. $NaOH$ in a 500 cc. tared flask. Add a few drops of pure mineral lubricating oil (to prevent foaming) and attach to the reductor contg. 20-30 cc. 1:1 H_2SO_4 . Heat strongly till the reaction begins and then keep heating slightly till the reaction is completed (about $1\frac{1}{2}$ hr.). Withdraw the flame, allow the app. to cool and the acid to enter the flask from the reductor, which is then washed 3 times with 20-30 cc. H_2O each time, the washings passing also into the flask. Dil. with H_2O , add concd. $NaOH$ to slight alk. reaction, keeping the flask cool all the time, bring to vol. and filter. Det. the N in 100 cc. by the hypobromite method in a Knop-Wagner nitrometer. The vol. of N obtained corrected to normal conditions and multiplied by 0.2512 gives percentage of ammoniacal + nitrated N in 0.5 g. fertilizer. Det. the insol. org. N by the Kjeldahl method upon residue B (left upon the filter after filtration of soln. A) dried in the oven; employ distn. or hypobromite. In the latter case first dil. the product from Kjeldahlizing with warming in a 500 cc. flask. (Liq-

uids containing high concns. of salts decompose the hypobromites with slow evolution of O after liberation of N. Diln. prevents such formation of O.) The author carried out his method upon pure nitrates and nitrated fertilizers with very good results.

ROBERT S. POSMONTIER

Volumetric determination of potassium and its application to the analysis of fertilizers. GUIDO AJON. *Giorn. chim. ind. applicata* 2, 422-6(1920).—The method is as follows: Take 25 cc. of a 2% soln. of KCl or K_2SO_4 equiv. resp. to 1.26% and 1.08% K_2O , put in a 150-200 cc. Erlenmeyer flask, add 50 cc. 2N tartaric acid slowly and while agitating, and then 25 cc. 0.5N NaOH. Shake for 5 min. Add little by little, while still agitating, 25 cc. 96% alc., and allow the whole to rest 6-8 hrs. Filter through a no. 400 Dreverhoff paper of 9 cm. diam., wash the ppt. 9 times with neutral 96% alc., decanting each time upon the filter, detach the filter and place in the pptn. flask. Add, while shaking, 0.1N NaOH in 96% alc. to alk. reaction (phenolphthalein indicator), allow to rest 5 min., during which time the pink color due to the indicator should not disappear. Then dissolve the ppt. by 0.1N aq. NaOH, titrating the excess with 0.1N HCl. The number of cc. of NaOH used up by the soln. of the bitartrate, multiplied by 0.00471, gives directly the amt. of K_2O present.

ROBERT S. POSMONTIER

Report on potash. T. E. KERR. *J. Assoc. Official Agr. Chemists* 4, 373-4(1921).—A modification of the perchloric acid method is proposed as a very satisfactory method of detg. potash in mixed fertilizers. Place 2.5 g. of the sample upon a 12.5 cm. filter paper and wash successively with portions of boiling H_2O into a 250 cc. flask until the washings amt. to 200 cc. Acidify with 5 cc. concd. HCl. While hot ppt. the sulfates by adding drop by drop in slight excess $BaCl_2$ soln. acidified with HCl. Cool, make up to mark, mix, and allow to settle. Transfer a 25 cc. aliquot to a porcelain evap. dish, add 30 cc. aqua regia and evap. to dryness. Dissolve in 20 cc. hot H_2O and add 5 cc. $HClO_4$ (d. 1.12). Evap. on a hot plate until copious fumes are evolved. Remove and run the liquid around the bottom of the dish. If solidification does not occur on cooling, continue the evapn. Take up the residue in 5 cc. H_2O and add a second portion of 5 cc. $HClO_4$. Evap. to dense fumes and remove so that the residue does not appear baked. In case it does, repeat the last operation. After cooling add 20 cc. 95% alc., stir and allow to stand 30 min. Decant through a Gooch crucible, and wash twice by decantation with 95% alc. containing 0.2% $HClO_4$. Transfer the ppt. with the same wash to the crucible and wash until the filtrate amts. to about 75 cc. Wash twice with alc.-ether (1:1) using 3-5 cc. each time. Dry for 30 min. at 120° and weigh. Dissolve the $KClO_4$ from the crucible with about 200 cc. hot H_2O and wash the asbestos with alc.-ether. Dry and weigh, taking the loss in wt. as $KClO_4$. S. G. SMERSON

Seed-coat injury and viability of seeds of wheat and barley as factors in susceptibility to molds and fungicides. ANNIE M. HURD. Bur. Plant Industry U. S. Dept. Agr., *J. Agr. Research* 21, 99-122(1921).—A study of (1) some of the factors, especially the phys. condition of the seed coat, which enable saprophytic fungi to attack the seed and of (2) the relation of mechanical injuries sustained by the seed coat to injury following seed treatment. The fungi used were *Penicillium* sp. and *Rhizopus*. Different varieties of wheat and barley were tested. The first portion of the paper contains no chem. data. These molds develop more slowly on stored wheat and barley which has been treated with $CuSO_4$ or HCHO than on equally moist untreated wheat. *Penicillium* is especially sensitive to HCHO. A break in the tests over the endosperm of wheat does not result in injury unless exposed to $CuSO_4$ for 1 hr. A similar break over the embryo results in its death after 3-5 minutes exposure. When seed coats are severely injured liming does not prevent extreme injury because the $CuSO_4$ enters such seeds quickly; with seeds only slightly injured a lime dip is effective. The outer layer of the seed coat may be broken over the embryo without injury resulting from $CuSO_4$, the inner layer being impermeable to the Cu. Stunted roots are characteristic of $CuSO_4$ injury.

The physical condition of the seed coat determines the extent of the injury on treating with CuSO_4 . Perfect seed coats are also an absolute protection against short exposures to strong HCHO solns. and are a partial protection against post-treatment injury after HCHO disinfection. Injury always occurred after exposures to satd. CuSO_4 solns. for periods of more than 6 hrs. and sometimes in less time. The temp. of the CuSO_4 soln. in which the wheat was immersed was a factor in the resistance to injury after long periods; the germination was poorer as the temp. increased. F. C. COOK

Combating sheep scab. A. NAGLER. *Fühling's Landw. Zig.* 70, 12-13(1921).—A report of expts. in which CO_2 was used. The method, results and cost are recorded.

F. M. SCHERTZ

BERKNER, F.: *Neue Wege der deutschen Landwirtschaft*. Berlin: Paul Parey 240 pp. M. 8.

KOZSCHNIK, F.: *Grundriss der landwirtschaftlichen Gewerbe (Landw.-chem. Technologie)*. 3rd Ed. Leipzig: Karl Scholtze. 1921. For review see *Deut. Zucker-ind.* 46, 180(1921).

PICK, S.: *Die künstlichen Düngemittel. Ein Handbuch für Fabrikanten künstlicher Düngemittel, Landwirte, Zuckerfabrikanten, Gewerbetreibende und Kaufleute*. 4th Ed. Wien and Leipzig: A. Hartleben. M. 7.50. For review see *Z. angew. Chem.* 34, Aufsatzteil 159(1921).

Fertilizer. ERLING BJØRNE JOHNSON and A.-S. NORTH WESTERN CYANIDE CO. Norw. 31,431, Dec. 6, 1920. Urea or urea salts are mixed with industrial CaNCN . They thereby lose their tendency to stick or become lumpy.

Fertilizers. D. LO MONACO. Brit. 159,481, Feb. 25, 1921. A fertilizer is obtained by treating humus with halogen gas. The treatment is carried out in a closed casing fitted with an agitator.

Fertilizers. SOC. D'ETUDES CHIMIQUES POUR L'INDUSTRIE. Brit. 159,853, Sept. 2, 1920. A fertilizer contg. N and sol. phosphate is obtained by adding while stirring powdered CaNCN to a H_3PO_4 soln. of free cyanamide and drying the granular product, H_3PO_4 contg. H_2SO_4 such as results from the treatment of $\text{Ca}_3(\text{PO}_4)_2$ with H_2SO_4 may be used.

Fertilizers. SOC. D'ETUDES CHIMIQUES POUR L'INDUSTRIE. Brit. 159,854, Sept. 2, 1920. A nitrogenous fertilizer is obtained by adding powdered CaNCN to a well stirred H_2SO_4 soln. of free cyanamide, and drying the product.

Nitrogenous fertilizer. NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB. Norw. 31,408, Nov. 22, 1920. Urea nitrate is mixed in finely divided state with CaCO_3 to bind the HNO_3 which is formed by the action of moisture on the urea nitrate. Cf. C. A. 14, 1404.

Superphosphates. OTTO ROSENTHAL. Norw. 31,045, Sept. 20, 1920. Dry P_2O_5 is mixed with raw phosphate and stirred with such an amt. of H_2O as is necessary to convert the pentoxide into H_3PO_4 .

Concentration of superphosphate by the dry process. COMP. DES PHOSPHATES DE CONSTANTINE. Norw. 30,634, May 3, 1920. The dried superphosphate is comminuted (but not ground) and sepd. by mechanical treatment (sifting, screening, centrifuging) into two products of different degrees of fineness, one coarse-grain product consisting of a concd. substance with a high % of $\text{CaH}_4(\text{PO}_4)_2$, and a poor product consisting of dust and containing much more gang and gypsum than the crude phosphate.

16—THE FERMENTATION INDUSTRIES

H. S. PAINE

Cold fermentation. G. FRIES AND R. HEUSS. *Z. ges. Brauw.* 43, 249-52, 257-80 (1920).—Healthy bottom-fermentation yeast which had been subjected to alk. treatment (see Lampitt, *C. A.* 14, 1351) was employed in large scale fermentations of 4% and 10% worts at temps. of 3-4°, instead of the usual temps. of bottom fermentation, viz., 4-10°. The results were unsatisfactory; fermentation occupied 1-3 days longer than usual, and although relatively large amts. of yeast were used for pitching, the yeast crops obtained were very small. Moufang's statement that low fermentation temps. improve the character of the beer produced was not confirmed. In the alk. treatment of the yeast, losses amounting to about 40% were incurred (cf. *loc. cit.*) J. S. C. I.

Power alcohol—proposals for its production and utilization in Australia. GERALD LIGHTFOOT. *Australia Inst. Sci. Ind. Bull.* No. 20, 104 pp. (1921); (A reprint of *Bull.* No. 6, 1917, with large addendum).—Some molasses is being made into alc. in Australia at present. Much more is available. Other raw materials which may be made available at a sufficiently low price are sorghum, sorghum grains, damaged wheat, cassava, and two native plants, the *Zamia* palm and the "grass tree." The *Zamia* palm (*Macrozamia spiralis*) is a slow-growing wild plant the swollen stem of which contains much starch. In exptl. work an av. yield of 18 imperial gallons of 95% EtOH per long ton of undried stems was obtained. Acid hydrolysis of the starch gave better yields than did diastase. The grass tree (*Xanthorrhoea*) has an elongated stem the core of which contains about 10% of sugars. The av. yield in expts. was 19 imperial gallons of 95% EtOH per long ton of fresh cores. The sources for the production of industrial alc. in other countries are reviewed. Changes in the design of gasoline engines and carburetors to adapt them to the use of alc. are discussed. Ordinarily an engine cannot be started cold on alc. unless some preheating or "hot spot" device is attached to the carburetor. However, it was found that by priming the cylinders with a little alc. and closing the throttle almost air-tight so that the amt. of air admitted to the cylinders was enough to produce only 25-35 lbs. per sq. in. compression the engine would start with a few turns of the crank. L. E. GILSON

Biochemistry of the mahua flower. G. J. FOWLER, J. D. E. BEHRAM, S. N. BHATE, K. H. HASSAN, S. MAHDIHASSAN, AND N. N. INUGANTI. *J. Indian Inst. Sci.* 3, 81-118 (1920).—Chem. and fermentation studies were made of the sugars in the mahua flower (*Bassia longifolia* and *B. latifolia*) with a view to its utilization as a source of industrial alc. Dextrose, levulose, maltose, sucrose, pentoses, and cellulose were identified, the total sugar being greatest when the flowers are ready to fall, at which stage it amounts in general to 60-70%. In the growing stages levulose is present in greater amt. than dextrose, but in the final stages the quantities approximate but do not become equal. Sucrose increases in amt. up to the shedding of the corolla, but after this and during storage it decreases relatively to invert sugar. Numerous enzymes were detected at various stages of growth of the flower; maltase, catalase, and oxidase were present throughout. Yields of alc. up to 90% of the theoretical were obtained by fermentation of a mash of the flowers with cultures of the natural yeast occurring in the flower, with the addition of such reagents as H₂SO₄ and NH₄ phosphate. By increased care in the cultivation, collection, and storage of the flowers a greater yield per tree, and a greater percentage of total sugar, and particularly of sucrose, should be possible of attainment. J. S. C. I.

Degeneration and treatment of yeast. R. HEUSS. *Z. ges. Brauw.* 43, 225-7, 233-4, 241-2 (1920).—A bottom-fermentation brewery yeast used in the production of dark, 4.5% war beers, was much improved by treatment with five times its vol. of water contg. 0.12% of free K carbonate for 15 hrs. at about 4°, followed by two washings with cold

water. Compared with the same yeast washed only with water, the treated yeast was much lighter in color, and its flavor was mild and free from bitterness. In the first fermentation after the treatment it produced a slightly less rapid attenuation than the control yeast, but in subsequent fermentations this difference was reversed. The break of flocculation of the treated yeast occurred at a later stage of fermentation than in the case of the control yeast. The beers produced were milder in flavor but possessed less palatableness than those produced by the control yeast. J. S. C. I.

Mashing experiments with a defectively modified malt. H. LÜERS and M. SCHNEIDER. *Z. ges. Brauw.* 43, 313-5, 321-4, 329-31 (1920).—A very badly modified malt contg. 22% of hard and steely corns and 15% of ungerminated corns, was subjected to lab. mashing tests by 15 different methods. The highest yield from grist of ordinary fineness was 75.62% of the dry substance and was obtained by a combination of cold pre-digestion, proteolytic digestion at 45°, and mash-boiling. J. S. C. I.

Determination of acidity (of worts) and titration in stages. H. LÜERS. *Z. ges. Brauw.* 43, 252-3 (1920).—A modification of Reichard's method of titration in stages (*C. A.* 11, 2013; 12, 2038) is described. J. S. C. I.

The production of several organic acids from sugar solutions. MAZZADROLI. *Centr. Zuckerind.* 1918, 257; *Z. Zuckerind. Tschoslow. Rep.* 44, 124 (1920).—M. has succeeded in producing, with specially grown cultures, lactic, acetic and butyric acids from the sugar in the beet juice. 100 g. of sugar yielded 60-80% of lactic acid, 10-20% of acetic acid and 1-7% of acetone. EtOH and the higher alcs. were present only in traces. Acetic acid was produced much more readily than lactic. The work on butyric acid was not completed. JOHN M. KENO

Report on wines. J. M. HUMBLE. *J. Assoc. Official Agr. Chem.* 4, 459-63 (1921).—Collaborative results on synthetic samples were unsatisfactory with the Rothenfusser method (*C. A.* 8, 1184) for glycerol. H. A. LEPFER

Sulfite spirit in 1920 (SIEBER) 23. Vinegar manufacture in the U. S. (HASSACK) 12. Alcoholic fermentation (KOSTYCHEV, ELIASBERG) 11C. Harvesting, storage and drying of barley (CLUSS, *et al.*) 12. Influence of temperature on various functions of yeast (ZIKUS) 11C. Determination of the total sulfurous acid in organic substances (PROBORS) 7. Alcoholic fermentation (KOSTYCHEV, *et al.*) 11A.

17—PHARMACEUTICAL CHEMISTRY

W. O. EMERY

Labdanum and the analytical characters of the oils of *Cistus Ladaniferus* L. and *Cistus Monspeliensis* L. ANON. *Roure-Bertrand Fils Sci. Ind. Bull.* [4] 1920, No. 2, 25-29.—Analytical data obtained by Pelletier and by Guibourt for labdanum are given, also data of a similar nature by Parry (*Chemistry of Essential Oils* Vol. 1, p. 503; cf. *C. A.* 5, 3879) and Masson (*C. A.* 6, 1286) for the oil distd. from the oleoresin. The author obtained 0.06% of a golden-yellow oil by steam distn. from the oleoresin extd. from *Cistus ladaniferus* L. This oil possessed the following characters: d_{20}^{20} 0.9053; $[\alpha]_D^{17}$ -12° 10'; $n_D^{12.5}$ 1.4800; acid no. 3.7; sapon. no. 22.37; ester no. 18.67; sol. in 90% alc. 1/2 to 5 vol. The extrn. of the distd. waters with petr. ether yielded an additional 0.02% of oil with the following characters: $d_{17.5}^{17.5}$ 0.9755; $[\alpha]_D^{17}$ -2° 40'; acid no. 18.67; sapon. no. 41.07; ester no. 22.40; sol. in 90% alc. 1/2 vol. and over. Data obtained by Schimmel and Co. (*Semi-Ann. Rept.* 1903, October, p. 81) for the oil obtained from *Cistus monspeliensis* are also given. A. G. DUMEZ

A summarized study of certain essential oils. ANON. *Roure-Bertrand Fils Sci. Ind. Bull.* 1920, [4] No. 2, 29-37.—Oil of *chenopodium*.—By steam distn. 0.7% of a

light yellow oil was obtained from chenopodium seed obtained from the Dutch East Indies. Its analytical consts. are: d_{44}^{20} 0.9763; $[\alpha]_D^{25}$ $\pm 0^\circ$; acid no. 0.98; sapon. no. 7.47; ester no. 6.54; sol. in 70% alc., 2 vol. and over. By extg. the distd. waters with petr. ether, an additional 0.35% of oil was obtained possessing the following characters: d_{44}^{20} 0.9843; $[\alpha]_D^{25}$ $\pm 0^\circ$; acid no. 0.93; sapon. no. 13.98; ester no. 13.06; sol. with difficulty in 70% alc. The optical rotation of these oils differentiates them from the American oil which has a $[\alpha]_D$ of -4° to $-8^\circ 30'$. Oil of *Skimmia laurole*.—A sample of oil distd. from the herb of *Skimmia laurole*, Hook. f. showed: d_{44}^{20} 0.8931; $[\alpha]_D^{25}$ $+4^\circ 28'$; acid no. 0; sapon. no. 82.13; sol. in 3 vols. of 90% alc. Oil of cinnamon from Annam: A sample gave: d_{44}^{20} 1.051; n_D^{20} 1.6090; $[\alpha]_D^{25}$ $-0^\circ 8'$; acid no. 2.8; aldehyde content 95%; sol. in 70% alc., 1. vol. and over. The odor of the oil is more pungent and less fine than that of oil of Ceylon cinnamon. It resembles a rectified Chinese oil of good quality in its characters.

A. G. DuMaz

Physical constants and characteristic derivatives of the principal constituents of the essential oils. ANON. *Rowe-Bertrand fils Sci. Ind. Bull.* [4] 1920, No. 2, 37-48.—A table is given showing the physical consts. and characteristic derivs. of the principal aldehydes found in essential oils, namely: aromadendral, myrtenal, dihydrocuminic aldehyde, citral, neral, phellandral, citronellal, *nor*-tricycloeksantalal, santalal, farnesal; formic, acetic, normal butyl, valeric, isovaleric, normal hexyl, normal octyl, normal nonyl, decyl, lauric, oleic, benzoic, phenylacetic, cuminic, cinnamic, salicylic, anisic, α -methoxycinnamic, and p -methoxycinnamic aldehydes; vanillin, piperonal, furfural and α -methylfurfural.

A. G. DuMaz

Essential oils of *Leptospermum flavescens* var. *grandiflorum* and *L. odoratum*. A. R. PENFOLD. *J. Proc. Roy. Soc. N. S. Wales* 54, 197-207(1921).—The crude oil obtained in a yield of 0.61% had the following consts. after being first cleared up with dil. NaOH soln.: d_{44}^{20} 0.9324; optical rotation -2.42 ; n_D^{20} 1.5048; ester no. 1.5 hrs. hot sapon. no. 7.2; ester no. after acetylation, 2 hrs. cold contact 24.95, 1.5 hrs. hot contact 40.98; insol. in 10 vols. 80% alc. It consists principally of levorotatory aromadendrene, eudesmene (slightly dextrorotatory) and an unidentified sesquiterpene alcohol, being distinct from the oil of *L. flavescens*, the chemistry of which will be communicated at a later date. The oil of *L. odoratum*, obtained in an av. yield of 0.75%, consists essentially of the following terpenic compds.: dextrorotatory eudesmol, levorotatory eudesmene, levorotatory aromadendrene, α pinene, β -pinene, together with small amts. of a rose odor alcohol, butyric and acetic acid esters, and phenols, one of which is probably identical with tasmanol.

W. O. E.

Monographs on floral odors. II. Jasmine odor. P. P. R. *Perfumery Essent. Oil Record* 12, 109-11(1921); cf. C. A. 15, 1962.—Besides enumerating the probable constituents of the natural "Otto," the article gives working formulas for jasmine, frangipani and bouvardia perfumes.

W. O. E.

Clove oil from clove stems. S. T. GADRE. *Perfumery Essent. Oil Record* 12, 115-7 (1921).—The oil distd. from a large consignment of stems from Zanzibar was obtained in an av. net yield of 4.5 lbs. per 100 lbs. of stems. It had a pale yellow color, and in aroma and quality was equal to the best English distd. clove oil, fulfilling all B. P. and U. S. P. tests. The d_{44}^{20} varied from 1.0522 to 1.0548; n_D^{20} 1.5345; sol. in 0.5 vols. 80% alcohol; total eugenol via Umney 93.09%, via Thom 83.53%; free eugenol via Varley and Bolsing 69.86%. Actual production costs are given.

W. O. E.

Standardization of disinfectants. FREYMUTH. *Pharm. Zig.* 66, 266(1921).—A discussion.

W. O. E.

Lead in tube preparations. K. NEUKAM. *Chem. Zig.* 45, 301-2(1921).—The investigation included both filled and unfilled collapsible tubes having a Pb content up to 96%. Many of the tinned Pb tubes were found to be imperfectly coated, while

tubes filled with various cosmetic and dental creams showed more or less advanced corrosion, the contents showing the presence of Ph in widely varying amts., from 0.003 to 0.017% for dentifrices, 0.012 to 0.266% for face creams, 0.01% for shaving cream, 0.007 for meat ext., lanolin 0.003%, borated lanolin 0.02%, depending among other factors upon the age of the product.

W. O. E.

The effects of prolonged heating and of aqueous extraction on opium. ALEX. M. MACMILLAN AND ALFRED TINGLE. *Am. J. Pharm.* 92, 810-15(1921).—Results show a steady loss of wt. when opium is heated, no sign of constancy being observed even after heating for 576 hrs. at 98-100°. One sample showed a loss of more than 1% in the period 192 to 576 hrs. contrary to the results recorded by Annett and Hardayal Singh (*C. A.* 13, 361), who claim to have obtained practically const. wts. after 192 hrs. Results also point to a sharp difference between Persian and Indian opiums. The former lost more than 30% of its morphine on being heated for 96 hrs., but lost no more when the heating was continued to 288 hrs. The Indian opium lost only about 3% of its morphine on being heated for 192 hrs. but lost progressively and heavily till it had lost 30% on 576 hrs. heating, contrary to Annett and Hardayal Singh, who claim to have found an increase in the morphine content from 264 to 288 hrs. heating. With the exception of losing its water of hydration during the first day of heating, no appreciable change could be detected when the free alkaloid morphine was heated for 8 days. As shown previously by others, water alone does not completely ext. morphine compds. from opium. It was found that 0.17% of morphine (nearly 2% of the total originally present) was not extd. even under somewhat drastic treatment and that the destruction of morphine by prolonged digestion of the ext. was 0.91% (more than 9% of the total originally present).

W. G. GAESSLER

Automatic separator for use in essential oil distillation. H. E. WATSON. *J. Indian Inst. Sci.* 3, 15-8(1920).—An automatic separator for immiscible liquids, adapted particularly to the sepn. of essential oils from aq. distillates, consists of a cylindrical vessel provided with a float kept in a vertical position by means of a stem which passes through a hole in the center of the cover of the vessel and terminates at the bottom in a conical needle valve forming the outlet for the heavier liquid. The float is weighted to take up a position of equil. at the surface sepg. the two liquids, and the distillate is introduced horizontally into the vessel at about this point. A rise in the level of the heavier liquid lifts the float and opens the needle valve, thus allowing a discharge of the liquid until the level has again fallen to the normal position. The lighter liquid collects in the upper part of the vessel until it reaches the level of a discharge pipe, through which it continuously flows off at a rate proportional to the inflow of the distillate. A separator of this type about 8 in. in diam. and 10 in. deep is capable of dealing with about 100 lb. of distillate per hr.

J. S. C. I.

Investigations on musk root oil. Inversion of bicyclic and tricyclic sesquiterpenes. MARTIN BAUER. Schlesische Friedrich-Wilhelms-Universität, Breslau. *Diss.* 1915, 36 pp.—The following compds. were established as present in the oil: (1) a mixt. of chiefly cyclic but with admixed aliphatic hydrocarbons, of compn. $C_{15}H_{24}$, boiling between 145° and 155°; (2) a new sesquiterpene, *sumbulene*, $C_{15}H_{24}$, 120-130°, $d_{20} 0.8999$, $n_D 1.49618$ and $[\alpha]_D 10^\circ 21'$. It is bicyclic with two double bonds and yields cadinene dihydrochloride on treatment of its cold ethereal soln. with dry HCl; (3) a dextrorotatory sesquiterpene identical with that found by Deussen in West Indian sandalwood oil. Treatment with HCl gave cadinene dihydrochloride, which in turn yielded levorotatory cadinene; (4) a mixt. of secondary and tertiary alcohols of compn. $C_{15}H_{26}O$ and $C_{15}H_{24}O$ boiling at about 160°; (5) an ester, $C_{15}H_{24}O_2$, which on saponification gave a solid fatty acid (probably angelic acid). This ester is perhaps admixed with some lactone of the same compn.

ALBERT R. MERTZ

Melting point of acetylsalicylic (o-acetoxybenzoic) acid. G. CAPPELLI. *Giorn. chim.*

ind. applicata 2, 291-300(1920).—The generally accepted m. p. of acetylsalicylic acid (*A*) is 135°. C. tested a number of samples of com. products and found that the m. ps. were not concordant among themselves, and that many were much below 135°. Tsakalotos believes that the variation of m. p. of *A* is due to presence of salicylosalicylic acid formed by partial decompn. of *A* at the m. p. (with loss of AcOH), and that the rigorous detn. of the m. p. is impossible. Francois believes that the detn. of the exact m. p. of *A* is possible if the substance is introduced into the heating bath only when the latter is at a temp. not more than 5° below the m. p., and the detn. does not occupy over 5 min., the m. p. is 132°, according to him. C., as a result of his expts., drew the following conclusions: (1) The detn. of the m. p. of *A* is possible and is a measure of the purity of the latter. (2) The m. p. of *A*, detd. in capillary tubes, may be carried out without fear of formation of salicylosalicylic acid. If this transformation took place during fusion, it would be inconsistent with the fact made clear by C. that many samples of crude *A* and *A* crystd. from anhydrous solvents had m. ps. either exactly corresponding or very near to 135°. (3) Com. qualities of *A* having m. ps. between 124° and 130° were very probably crystd. from mixed solvents, *e. g.* acetone-H₂O, EtOH-H₂O. The lowering of the m. p. of samples of *A* crystd. from these mixed solvents was confirmed by C.'s expts. It is due to adsorption of H₂O. Grades having these m. ps. must be considered as not corresponding in their desirable properties with the official *A*. The partial sapon. which takes place with com. samples on lapse of time is due doubtless to presence of H₂O. C. observed that the least recent of his lab. samples had the odor of AcOH and contained free salicylic acid. (4) The m. p. of pure *A*, detd. in capillary tubes, is 135° by the Hans-Meyer method (clear and transparent liquefaction), and 132° by the Reissert method (m. p. taken at the moment of initiation of fusion of the first particles).

ROBERT S. POSMONTIER

Extracts prepared according to the official Italian pharmacopoeia. BERNARDO ODDO. *Giorn. chim. ind. applicata* 2, 485-91(1920).—The ext. treated in this article is the hydroalcoholic ext. of horned rye infected with ergot. The topics comprize: qual. tests such as external characteristics, solubilities, behavior of the aq. soln., identification of the active principles (1) ergotoxine ($=C_{28}H_{40}O_8H_2$); (2) *p*-oxyphenylethylamine ($=C_{12}H_{15}ON$); (3) agmatin ($=C_8H_{14}N_4$); (4) β -aminoethylglyoxaline ($=C_6H_9N_2$); also quant. tests, including the following detns.: dry residue at 100°, ash and its alkalinity, free acids, active principles. Tables are appended giving data on external characters, soly. in various solvents, general and sp. reactions of the H₂O-sol. portion, and analytical compn.

ROBERT S. POSMONTIER

The origin, development, and value of the thalleioquin reaction. WM. BEAMONT HART. *J. Soc. Chem. Ind.* 40, 72-3T(1921).—Although the green coloration produced by the addition to a quinine soln. of Cl or Br water followed by NH₄OH is a sensitive test for quinine, attempts to make this a quant. colorimetric method have not been satisfactory. The shade of color produced depends on the quantity of Br, the time of action of Br before the addition of NH₄OH, and the deterioration of color on standing. Excess of Br and its prolonged action are detrimental to the production of the thalleioquin reaction, 6 atoms of this halogen per mol. of quinine for 1 min. being ample for strongest color depth. Using this ratio the reaction begins at a diln. of 1:200,000 parts of quinine. As a qual. reaction, by making a few preliminary trials it can be made very sensitive, the limit being 1:250,000 in a depth of liquid 2 1/4 in. For quant. work, with so many variables, no credence of even approximate accuracy can be assigned to the reaction except under very strict conditions and in very dilute solns. The Br absorption of quinine in aq. soln. may be used as a quant. test allowing the Br to act for 5 min. and titrating the excess Br with Na₂S₂O₃ after the addition of KI. 1 cc. 0.1 *N* Br = 0.0081 g. quinine.

S. G. SMITHSON

Bromural. R. YOSHITOMI AND K. WATANABE. *J. Pharm. Soc. Japan* No. 468,

125-30(1921).—Bromural is one of the drugs successfully manufd. in Japan since the war. Although the pure prepn. should m. 154°, the com. product m. 145-56°. The authors studied the causes for this discrepancy in m. p. According to Nishizaki and Nishihara (*Ibid* no. 404), iso-amyl alc. is oxidized to isovaleric acid and brominated, in presence of P, to α -monobromoisovaleryl bromide, which is then condensed with urea to form α -bromoisovalerylurea; this is bromural. Amyl alc., however, obtained by distn. from fusel oil is very difficult to free from optically active amyl alc., which on oxidation goes over to $\text{CH}_3\text{EtMeCO}_2\text{N}$. During bromination, therefore, besides bromural, CBrEtMeCONNCONH_2 will be formed. Even if absolutely pure isovaleric acid is used for bromination, according to the temp. and amt. of Br, isovaleryl bromide is apt to be formed, which on condensation with urea becomes isovalerylurea. The authors prepd. pure bromural and isovalerylurea and detd. the m. ps. of various mixts. of these two. A chart is given to show the effect of this impurity on the m. p. of the bromural. The result shows that there are 2 identical m. ps. of the mixts. if bromural content is between 65% and 100%, that is between m. ps. 139 and 152°. Both 95% and 67% mixts. m. 147°, while both 100% and 75% m. 152°. M. p. alone, therefore will not show the purity of the bromural when isovalerylurea is present as an impurity.

S. T.

Manganese in fox-glove leaves. WEBSTER. *Ber. pharm. Ges.* 30, 376; *Pharm. J.* 105, 533(1920).—W. confirms Burmann's statement (*C. A.* 6, 410) that Mn is a const. constituent of *Digitalis purpurea*; he obtained 0.00094 to 0.00812 g. from 100 g. of dried leaves. Contrary to B., he also finds Mn present in *D. ambigua* and *D. lutea*.

S. WALDBOTT

Rapid disappearance of oil from the blood. H. BUSQUET AND C. VISCHNIAC. *J. pharm. chim.* 22, 190(1920); *Pharm. J.* 105, 379(1920).—When oil in a dose of 2-3 g. per kg. body wt. is given to a dog by intravenous injection, no trace of the oil or its fatty acid can be detected 15 min. after injection. This rapid disappearance explains the relative harmlessness of oil when used as a vehicle for intravenous medication.

S. WALDBOTT

The supplement to the French Codex of 1908. ANON. *Pharm. J.* 105, 301-2(1920).—Descriptive.

S. WALDBOTT

The French pharmacopeia supplement. G. P. FORRESTER. *Schweiz. Apoth. Ztg.* 58, 677-80(1920).—A detailed discussion.

S. WALDBOTT

Cascarilla bark. KUNZ-KRAUSE. *Arch. Pharm.* 258, 183; *Pharm. J.* 105, 533(1920).—Detn. of the compn. of the mineral constituents was the principal object sought. The aq. ext. of a genuine bark dried over H_2SO_4 was 4.34% (4.80 to 13.25% in previous records). Ash, 11.85%, was obtained after repeated treatment with NH_4NO_3 . Sol. in H_2O : K, Na, Cl, SO_4 ; sol. in HCl: CaCO_3 , AlPO_4 , FePO_4 ; insol. in HCl: SiO_2 . The tincture (1 in 5, with 70% EtOH) yielded about 3% of dry ext. Cl in ash was 0.516% and entirely H_2O -sol. Aq. solid exts. of the bark contained 8.95 and 15.01% KCl in form of cubes. Thus, cascarilla bark may be regarded as a "chloride drug," while henbane belongs to the "nitrate drugs" and stramonium has an intermediate position.

S. WALDBOTT

The use of poke root in medicine. E. M. HOLMES. *Pharm. J.* 105, 417(1920).—"There seems to be sufficient evidence that the root, besides possessing emetic, cathartic and cholagogue properties, acts as a discutient for tumors." Perhaps its "saponin may possess special hemolytic actions."

S. WALDBOTT

The use of poke root in medicine. U. AYLMEYER COATES. *Pharm. J.* 105, 454(1920).—The monograph of F. Peyre Porcher, *Resources of the Southern fields and forests, etc.*, Charleston, S. C. 1863, dwells in detail on the medicinal virtues of poke root. The prepn. of a crimson dye, the "solferino" color of 1862, from it is also described.

S. WALDBOTT

HUMPHREY, JOHN: *Drugs in Commerce*. London: Isaac Pitman & Sons. 116 pp. 3s. net. For review see *Pharm. J.* 106, 347(1921).

KISSLING, RICHARD: *Handbuch der Tabakkunde, des Tabakbaues und der Tabakfabrikation*. 4th Ed. revized and enlarged. Berlin: Paul Parey. M. 47.50. For review see *Z. angew. Chem.* 34, Aufsatzteil 160(1921).

MARTINDALE, W. HARRISON AND WESTCOTT, W. WYNN: *The Extra Pharmacopoeia*. 17th Ed. Vol. II. London: H. K. Lewis & Co. 688 pp. 17s. 6d. net. For review see *Pharm. J.* 106, 309(1921). Cf. *C. A.* 14, 2680.

Synthetic drugs. *CHEMISCHE FABRIK AUF ACTIEN VORM. E. SCHERING*. Brit. 158,558, Jan. 25, 1921. A double compd. of dimethylaminodimethylphenylpyrazolone and diethylbarbituric acid is obtained by melting the two components together preferably in mol. proportions. The product has strong analgetic properties.

Medicated milk preparations. F. STROHR. Brit. 159,877, Mar. 1, 1921. In a compn. made by condensing milk with reducing sugars, such as lactose, dextrose or maltose, cane sugar, and ferrisaccharates, the cane sugar is replaced in part by invert sugar. The mixt., before heating *in vacuo* to reduce the ferrisaccharate, is neutralized or rendered slightly alk. by the addition of alk. hydroxides or carbonates. Other medicaments may be added, and the milk dried to a powder.

Preparation of liquid disinfectants free from harmful secondary actions. PAUL SÄXL. Ger. 317,185, Aug. 31, 1918. The substances obtainable by the process protected by 316,015 are distributed and left for some time in H₂O or aq. solns. of NaCl, sugar or the like. Example: 100 g. of the glass material or quartz sand activated in the manner described in 316,015 are mixed with 50 g. of a 4% NaCl soln. and made to act thereon for 48 hrs., the mixt. being shaken several times. The NaCl soln. thus obtained is claimed to be capable, even in 100-fold diln., of killing off large amts. of pathogenic germs. This soln. is stated to be serviceable for surgical and medicinal purposes, also for the sterilization of *drinking water*.

Disinfecting process. ARNOLD HAHN and MAX FEDERER. Ger. 322,739, June 29, 1911. The effectiveness of a disinfectant depends in a high degree on the form and the physical conditions in which it is used, and still more on the nature of the medium in which it is dissolved. When highly viscous aq. solns. of otherwise entirely indifferent substances are employed as solvents or carriers of disinfectants, a much higher bactericidal action is attained than when merely aq. solns. are employed. It is above all the derivs. of *laminaric acid* that are stated to be particularly suitable for the prepn. of highly effective solvents, more particularly the sodium-ammonium salt of this acid. Example: 50 kg. of a 10% soln. of NaNH₂ laminarate are mixed with 50 kg. of 2% aq. HCHO. The mixt. is immediately ready for use.

Conversion of immune substances obtained from special proteins into general protein charged with other immune substances. ELEKTRO-OSMOSE-A.-G. (Graf-Schwerin-Gesellschaft). Norw. 30,978, Aug. 9, 1920. From immune serum the euglobulin is sepd. out, preferably electroosmotically, dissolved in blood serum from normal not immunized animals, or preferably in a soln. of paraglobulin from blood serum of not immunized individuals. From this mixt. the euglobulin is again sepd. out electroosmotically, the product being a paraglobulin soln. charged with immune substances.

Double salt of glycerophosphoric acid and lactic acid. E. MACK, *CHEMISCHE FABRIK*, and LEO WEBER. Ger. 331,695, Apr. 14, 1920. Either the Ca salts or the ferric salts of glycerophosphoric acid and lactic acid, or the components of these salts, are made to act upon one another.

Compound of chloral with a phenol. OSCAR HINSBERG. Ger. 332,678, Apr. 15, 1917. Chloral (85 g.) and *p*-AcNHC₆H₄OH (50 g.) are made to act upon one another with or

without the addition of a solvent. The product has a sleep-inducing effect when taken in smaller doses than chloral and is entirely tasteless.

18—ACIDS, ALKALIES, SALTS AND SUNDRIES

F. C. ZEISBERG

Thomas Lynton Briggs. A. H. SABIN. *J. Ind. Eng. Chem.* 13, 489(1921).—An obituary. E. J. C.

Method of recovering sulfuric acid from sodium bisulfate. I. MOSCICKI AND W. DOMINIK. *Przemysł Chem.* 4, 17-23(1920).— NaHSO_4 is mixed with an equal quantity of sand or Na_2SO_4 , and the H_2SO_4 is then distd. off, only about 0.1% H_2SO_4 remaining in the residue. J. S. C. I.

Water-cooled chambers adopted in England. A. M. FAIRLIE. *Chem. Met. Eng.* 24, 796-9(1921).—F. gives an account of the history, construction (cuts) and operation of the Mills-Packard water-cooled H_2SO_4 chamber invented by W. G. Mills and C. T. Packard of Ipswich, England, in 1914. In the early part of 1921 there were 111 of these chambers installed or contracted for. The Mills-Packard chamber has the shape of a frustum of a cone, the curtains being supported by either steel or wood columns, and dipping into acid contained in a lead pan. The tower is cooled by water flowing from the top (which is either dished or raised) down the outside of the curtains, which are divided into 5 sections. At the base of each of these a lead trough and a gutter burned to the curtains encircle the chamber. The rim of each gutter is serrated in order to distribute the overflowing water uniformly over the wall of the next section. The advantages claimed are—(1) Chamber space required per unit of S burned reduced to from 1/2 to 1/3 the usual space. (2) Reduction in cost of chamber construction per unit of capacity for making acid is said to be from 30 to 40%. (3) A substantial saving in ground space. (4) Longer life of chambers. (5) Usually no housing required for chambers, except in very exposed places when wood framework is employed. (6) Niter consumption per unit of S burned no higher than for rectangular chambers. (7) Feasibility of combining one or more M.-P. chambers with rectangular chambers of existing plants, or with tower systems, to increase production capacity at small construction cost. (8) Low maintenance cost. None has to date been erected in the U. S. A. For patents see *C. A.* 8, 3621, 3841; 13, 1748, 2576.

E. G. R. A.

Substitution of lignite of San Giovanni Valdarno for coke with the Perrin sulfuric acid concentrating apparatus. ARRIGO LINARI. *Giorn. chim. ind. applicata* 2, 492-3(1920).—The author found that with the high war-time price of coke the substitution of lignite for it proved advantageous, without detriment to yield of H_2SO_4 . Certain modifications of the app. and precautionary measures in carrying out the processes were found necessary. ROBERT S. POSMONTIER

Provisional regulations regarding policing, safety, and health in the nitrate plants. AXON. *Caliche* 1, 241(1919).—Transcription of presidential decree revising police, safety and health regulations operative in the nitrate works. C. L. BURDICK

Cylindrical container for shipping Chile nitrate. PEDRO LIRA ORRIGO. *Caliche* 1, 252, 286, 321(1919).—The process of prep. nitrate for shipment by hydraulic compression at 625 kg. per sq. cm. and fastening of wood strips on the outside of the blocks is described. The nitrate cakes prep. by the press have strength equal to mortar. C. L. BURDICK

Iodine corrosion in the evaporation of nitrate liquors. J. R. LORTSCH. *Caliche* 1, 445(1920).—Corrosion caused by liberated I presents one of the chief difficulties encountered in the application of multiple effect evapn. of the solns. obtained in nitrate

leaching. This difficulty has been successfully overcome by the addition of alkalis such as NaOH or CaO to the solns. to be treated. C. L. BURDICK

The question of borax in Chile nitrate. B. DIAZ OSSA. *Caliche* 1, 409(1920).—The occurrence of borax is discussed, its behavior in the treatment of nitrate ores, the means of avoidance in the finished product and the methods of detn. The 2 chief means proposed or already in use for the elimination of borax are (1) treating the solns. with an acid or SO_2 in the presence of iodates for the liberation of the almost insol. H_3BO_3 and (2) treatment with CaCl_2 at a boiling temp. to form insol. $\text{CaB}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. C. L. BURDICK

Nitrate technology, comments on its theory. E. ALMEYDA ARROYO. *Caliche* 1, 423(1920).—Controversy with Hobbsbawn as to the relative merits of their proposed processes. C. L. BURDICK

The problem of nitrate is solved. E. ALMEYDA ARROYO. *Caliche* 1, 448(1920).—An optimistic view of the author's own process. C. L. BURDICK

Analysis of nitrate manufacture. E. ALMEYDA ARROYO. *Caliche* 2, 7-13(1920).—More generalities regarding the proposed hot centrifugal process. C. L. BURDICK

Nitrate technology. J. B. HOBBSBAWN. *Caliche* 2, 14-5(1920).—Answer to Almeyda. C. L. BURDICK

Analysis of the manufacture of nitrate. E. ALMEYDA ARROYO. *Caliche* 2, 52-6(1920).—A general discussion of the characteristics of the solns. and mother liquors utilized in nitrate extrn. as influenced by the content of the solns. in various Na salts. C. L. BURDICK

Nitrate technology. E. ALMEYDA ARROYO. *Caliche* 2, 73-6(1920).—Controversy with Hobbsbawn and Lortsch as to the relative merits of their proposed processes. C. L. BURDICK

Observations on nitrate technology. J. B. HOBBSBAWN. *Caliche* 1, 321(1919).—Exception is taken to Perroni's contention that no single process is applicable in the entire nitrate industry. It is contended that if the grade of caliche worked can be dropped to 8% a really workable process would revolutionize the entire industry. C. L. BURDICK

Observations on nitrate technology. J. B. HOBBSBAWN. *Caliche* 1, 364(1920).—Detailed development of the author's ideas regarding the most economical nitrate recovery process of the future. He believes the process will embody (1) fine grinding of the caliche, (2) cold or tepid leaching together with classification of sands and slimes, (3) counter-current washing of sands and filtration of slimes, and (4) multiple effect evapn. followed by crystn. of the nitrate. C. L. BURDICK

Combination process for the manufacture of nitrate. MANUEL OSSA RUIZ. *Caliche* 1, 393(1920).—The process consists in (1) leaching the caliche at atm. temp., (2) solar concn. and evapn. to give a mixt. of salts and (3) redissolving the nitrate by hot solns. followed by cooling and crystn. C. L. BURDICK

The Haber process at Oppau. J. R. PARTINGTON. *J. Soc. Chem. Ind.* 40, 99-101R(1921).—A description of the equipment and method of operation of the Haber synthetic NH_3 plant at Oppau. W. H. ROSS

Government fixed nitrogen research. R. C. TOLMAN. *Chem. Mct. Eng.* 24, 595-9(1921).—A review of the present activities and tentative future plans of the Fixed Nitrogen Research Lab., American Univ., Wash., D. C. W. H. ROSS

The manufacture of Glauber's salt. W. HÜTTNER. *Chem. Ztg.* 45, 313-5(1921).—The demand for HNO_3 for explosives during the war produced large quantities of NaHSO_4 . This was later heated with NaCl forming Na_2SO_4 and HCl. The high prices paid for the two products caused a great expansion in the industry. The decrease in demand for HNO_3 since the armistice and its production by fixation of atmospheric N have caused a small resumption of the $2\text{NaCl} + \text{H}_2\text{SO}_4$ process. Production by crystn.

from NaCl and MgSO_4 in refuse liquors of the potash industry has also been revived. The residuum consists of rock salt and kieserite. Large quantities of this residuum have accumulated. From these $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ crystallizes during cold weather. As high as 14% has been found. Warm water (40–50°) is sprinkled over the piles and the resulting soln. caught in reservoirs. From here it is pumped to crystg. flats of wood or iron. The salt crystallizes at about –5°. The raw soln. averages 68.4 H_2O , 21.1 NaCl, 9.1 MgSO_4 , 1.3 MgCl_2 . From 1000 kg. of soln. are obtained 135 kg. of the salt. A sp. gr. of 1.29 for the raw soln. has been found most successful. The old method depended upon natural cold. At present artificial refrigeration is employed and salt solns. are used. The crude product is freed from iron by CaCl_2 and $\text{Ca}(\text{OH})_2$. Crystal sizes demanded are produced by the amt. of agitation afforded. Drying and storing are accomplished as far under 25° as possible. Calcining is done in perforated buckets which pass through a bin of the salt; drainage is allowed and the salt is conveyed to drying app. The Na_2SO_4 replaced by NaCl while in molten condition is recovered by centrifuging. The residues from NaCl smelting of S-bearing ores is suggested as a new source of Glauber's salt.

G. R. JACKSON

American enterprise makes "rare earth" a misnomer. G. A. PRITCHARD. *Chem. Age* (N. Y.) 29, 45–46 (1921).—Attention is called to the fact that a production of 15000 tons per year of the minerals zircon, ilmenite and rutile makes the term "rare earth" as applied to these minerals a misnomer. The high grade character of the Florida deposits of zircon and ilmenite is especially mentioned. The properties and uses of these 3 minerals are discussed.

P. E. B.

Asbestos in 1919. J. S. DILLER. U. S. Geol. Survey, *Mineral Resources of U. S.*, 1919, Part II, 299–307 (preprint No. 21, published May 14, 1921).

E. H.

Industrial production of helium. I. CAMILLO PORLEZZA. *Giorn. chim. ind.* 2, 638–47 (1920).—The following topics are taken up: application of He in aeronautics, first proposals and practical expts. looking to industrial prepn., industrial prepn. of He in the U. S. and Canada. II. *Ibid* 687–91. Expts. in Italy and other applications of He, researches upon Italian natural gases and Italian scientific literature concerning He. Illustrations and sketches of app. are given and descriptions of processes employed.

ROBERT S. POSMONTIER

Artificial masses for toys. E. J. FISCHER. *Kunststoffe* 11, 33–5, 51–3 (1921).—The prepn. of plastic masses from cellulose, its esters, starch, etc. is reviewed. C. J. WESR

Recovery of ammonia as a by-product in the sugar industry (RUEFF) 28. The equilibrium between nitric oxide, nitrogen peroxide and aqueous solutions of nitric acid (BURDICK, FREED) 2. Recovery of ammonia from the gases escaping during saturation (RUEFF) 28. Inorganic chemical industry in Germany during the war (GALLI) 13.

ALLEN, A. W.: The Recovery of Nitrate from Chilean Caliche. London: Chas. Griffin & Co. 6s. For review see *Mining Sci. Press* 122, 732 (1921).

FOWLER, GILBERT J.: The Conservation of Nitrogen with Special Reference to Activated Sludge. Bangalore, India: Indian Inst. of Science. 52 pp. For review see *Eng. News-Record* 86, 865 (1921).

Hydrochloric acid. E. NICCOLI. *Brit.* 159,869, Jan. 25, 1921. In the combustion of H and Cl to form HCl, the furnace chamber, which may have a square or circular cross-section, is provided with a safety flame fed with an independent supply of H. H at a pressure varying from 5 to 20 mm. of H_2O is supplied to the furnace through a pipe and lighted by a Bunsen flame; the necessary air is admitted through a pipe.

Cl also at an adjustable pressure is then introduced through a pipe, and when the combustion proceeds normally the air supply is stopped. The branches of the H and Cl supply-pipes are made of quartz or other fireproof material and are arranged so that the H stream faces the Cl stream to insure intimate mixing of the gases. The furnace is closed by means of a very light cover cemented to the walls with a thin layer of fireproof glazing material, so that little resistance is offered in the event of an explosion. The HCl produced is withdrawn through a quartz conduit.

Concentration of sulfuric acid. JOHN WALKER LETCH. *Norw.* 31,437, Dec. 13, 1920. Three processes not new in themselves are combined in to a three-stage process for the purpose of producing a 96-98% acid. The dil. acid is first boiled down in open Pb vessels to 70-80%, then concd. in quartz or porcelain vessels in a closed chamber to 85-90% and finally passed through a special closed Fe vessel, where it is heated at the inlet to at least 260° and at the outlet to not more than 325°, and concd. to 96-98%. The vapors of the second concn. are collected and used to preheat the dilute acid, while the hot vapors of the last concn. are passed into a tower, where they mingle with the dil. acid and heat the latter, partially concg. it.

Highly concentrated nitric acid. E. BERGVE. *Can.* 211,255, May 3, 1921. N_2O_4 , O and water are caused to react at a pressure of 20 atm. and at about 70° to produce concd. HNO_3 .

Ammonia; oils. P. BRAT. *Brit.* 159,194, Feb. 17, 1921. NH_3 is obtained from peat by a process of the kind described in 157,745 (C. A. 15, 1971) and 157,746 (C. A. 15, 1971) by heating the peat with basic substances such as Ba, Sr, Mg or Al_2O_3 or Na aluminate or mixts. of these substances together with H_2O in an autoclave at 170-200° under at least 6 atm. pressure. By continuing the heating in some cases after the sepn. of the H_2O the hydrocarbons can be obtained from the peat without the necessity of adding a hydrocarbon of high b. p. Such further heating may be at 200-700° or over. If a hydrocarbon of high b. p. such as pitch is added to the residue of the peat, the hydrocarbons are obtained by heating up to about 500°.

Ammonia; salts of acids obtained from peat. P. BRAT. *Brit.* 159,193, Feb. 17, 1921. In the production of NH_3 by the treatment of peat in an autoclave under pressure of at least 6 atm. at 170-200° in presence of an alk. reagent as described in 157,745, (C. A. 15, 1971), 157,746, (C. A. 15, 1971), and 159,194, preceding pat.) salts of water-sol. acids are obtained in the liquid in the autoclave. The peat residue containing this liquid is subjected to pressure, osmosis, or luviation and the salts of other metals are obtained by double decompn. The Pb and Hg salts are insol. The acids and their salts can be used as disinfectants or for therapeutic purposes. In addition to the basic reagents mentioned in the specifications mentioned above, KOH and borax may be employed for the ammonia-producing reaction.

Synthetic ammonia. NORSK HYDRO-ELEKTRISK KVAELSTOFKATIESELSKAB. *Brit.* 159,878, Mar. 1, 1921. Catalysts for the synthesis of NH_3 from its elements consist of Fe, Co, Cr or Mn, activated by the addition of cyanides of the alkali or alk.-earth metals; a pressure of 80 atm. and temp. of 375° are mentioned as suitable for the reaction with Fe and KCN as the catalyst. Cf. C. A. 15, 929.

Synthetic ammonia. L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDÉS G. CLAUDE. *Brit.* 158,849, Feb 2, 1921. The residual gases from the synthesis of NH_3 under high pressure are re-circulated through the series of catalyzing app. by returning them to the inlet to the last cylinder of the compressor. The accumulation of inert gases, such as A, is prevented by periodically allowing the residual gases to escape to the atm. Cf. 130,086 (C. A. 14, 104).

Cyanamide. WARGONS AKTIEBOLAG and J. H. LIDHOLM. *Brit.* 158,806, Jan. 18, 1921. Solns. of cyanide contg. little dicyanodiamide are obtained by adding CaNCN in portions to H_2O and continuously supplying CO_2 as described in 151,583, (C. A. 15, 678)

and so regulating the supply of the CaCN_2 and CO_2 that the alkalinity of the soln. does not exceed 0.5 N. Limited cooling may be employed, but the temp. should be not less than 30° .

Production of cyanamide solutions from crude calcium cyanamide. HEINRICH DANNREIL and ELEKTIZITÄTWERK LONZA, AKT.-GES. Ger. 302,495, Mar. 13, 1917. CO_2 is passed into an aq. suspension of CaCN_2 . In addition to the intended reaction: $\text{CaCN}_2 + \text{H}_2\text{O} + \text{CO}_2 = \text{CaCO}_3 + \text{H}_2\text{CN}_2$, there occurs a carbonation of the free lime, more or less of which is always present in crude CaCN_2 . The reaction must proceed in the cold, since otherwise a portion of the cyanamide would be converted into NH_3 and another portion into dicyanodiamide. At the same time a considerable amt. of heat is liberated, which must be removed by rapid cooling. Expts. are stated to have shown that the injurious action of heating may be considerably reduced by effecting the carbonation of the free lime in the dry state before it is passed into H_2O . As in this process the soln. becomes less alk., the formation of dicyanodiamide, which depends on the degree of alkalinity, is lessened.

Alkali cyanides. B. BROCH. Norw. 31,292, Oct. 25, 1920. Pure dry N is forced through an alkali metal bath heated to or above the b. p. The mixt. of N and alkali metal vapor formed is passed under pressure through C heated to above the m. p. of the alkali cyanide. Alkali carbide then forms, which in the nascent state enters into reaction with the N, forming alkali cyanide.

Ammonium chloride. O. L. CHRISTENSON and B. A. HEDMAN. Brit. 159,817, Feb. 24, 1921. In the coking of coal or other fuels, the N present is recovered as NH_4Cl by adding an alkali or alk.-earth chloride and SiO_2 to the furnace charge and heating in the presence of steam. In an example, salt and silica, finely ground and mixed into a pulp with H_2O are spread uniformly on the fuel before it is coked. The NH_4Cl may be sep'd. from the distn. gases either by cooling to a temp. just above that at which the H_2O and other vapors condense, when the NH_4Cl is obtained in a dry condition, or by cooling and condensing all the products together and crystg. the resulting gas liquor. The crude NH_4Cl , which contains a small amt. of phenols, is particularly suitable as a *fertilizer*. The presence of alkali or alk.-earth silicates in the coke reduces the consumption of flux when it is used in blast-furnace processes.

Ammonium sulfate. GUSTAF HENRIK HULTMAN. Norw. 30,822, June 21, 1920. In order to convert $(\text{NH}_4)_2\text{SO}_4$ into $(\text{NH}_4)_2\text{SO}_4$, it is treated with air or O or a mixt. of both, the amt. of moisture necessary for oxidation being previously added to the air.

Dichromates. SOC. IND. PROD. CHIM. Norw. 31,323, Nov. 1, 1920. Alkali chromates are dissolved in an amt. of H_2O insufficient for soln. at 15° , org. solvents added and partial or complete conversion into dichromates is effected by means of CO_2 .

Alkali and alkaline-earth sulfides. L. P. BASSET. Brit. 159,202, Feb. 19, 1921. Sulfates of the alkali and alk.-earth metals are mixed with the necessary amt. of coal which is preferably non-bituminous, and converted to the corresponding sulfides by means of a reducing flame obtained by the combustion of coal, in the form of an impalpable powder, with heated air in such quantity that only CO is formed. Oils or hydrocarbons may be atomized and used in place of the pulverized coal, and steam may be admitted with the air, the latter being introduced in sufficient quantity to burn the H liberated. The reduction may be carried out in a reverberatory furnace such as is described in 140,096 but preferably it is performed in a rotary furnace having its axis slightly inclined to the horizontal. Hot air is injected at the lower end together with the oil or powdered coal and the mixt. of sulfate and coal is fed in continuously at the upper end. The combustible gases leaving the furnace are utilized to heat the air injected with the coal or oil.

Sodium bicarbonate. NITROGEN CORPORATION. Brit. 159,895, Mar. 8 1921. In the ammonia-soda process, NH_3 and CO_2 are employed under pressure, preferably at 100

atm., or in the liquid condition, and the low temp. produced by their expansion is utilized to cool the liquors in the absorbing and carbonating towers. After passing through the expansion valves and cooling coils, the CO_2 is dild. to the extent of 40-45% with N, in order to moderate the reaction. The N is supplied under pressure and preferably in "pulses," and is collected for re-use after passage through the carbonating towers. CO_2 may be used to cool the absorbers, but NH_3 is more suitable for this purpose since in the event of leakage, the reaction in the tower is not affected.

Isolation of chromium salts. SOC. IND. PROD. CHIM. Norw. 31,374, Nov. 15, 1920. The caustic alkalies are sepd. from their mixts. with chromates and other salts, if any, by means of org. solvents. Caustic alkalies, carbonates, aluminates and silicates of Na and K are sepd. from alkali chromates by means of CO_2 .

Stannous chloride. J. J. COLLINS. Brit. 159,659, Dec. 10, 1919. Crude Sn is granulated or divided and introduced together with Cl and H_2O into a vessel containing SnCl_2 or SnCl_4 , the Sn being maintained in excess so that SnCl_2 is formed. Pure Sn may be obtained from the SnCl_2 by known methods so that the process may be used for refining Sn.

Alumina from clay. WILHELM DAN BERGMAN. Norw. 30,846, June 28, 1920. Clay is decomposed with an acid soln. and the $\text{Al}(\text{OH})_3$ pptd. by means of an alk. soln. for which purpose the solns. forming in the electrolysis of a neutral sulfate soln. are used. From the acid anode liquid the neutral sulfate is sepd. by crystn., said liquid then being used to decompose the clay. The resultant Al soln. is sepd. from the insol. constituent (silicic acid) and pptd. with the alk. cathode liquid. $\text{Al}(\text{OH})_3$ then seps. off and neutral sulfate re-forms, which is again returned to the process.

Titanium and vanadium compounds. B. P. F. KJELLBERG. Brit. 159,532, Oct. 20, 1919. Materials containing Fe, Ti and V are treated with an acid such as H_2SO_4 to dissolve the metals, and the soln. is evapd. to dryness. The residue is dissolved in H_2O and the soln. heated to ppt. the Ti, the remaining soln. being evapd. to dryness. The second residue is heated to decompose the Fe salt and then treated with H_2O with or without an acid or alkali to dissolve the V. Alternatively, when comparatively little Ti is present the original material may be treated with dil. acid so as to dissolve the bulk of the Fe and V, leaving a Ti concentrate which can be treated as above described to sep. the Ti as a ppt., and the remaining soln. is added to the soln. already obtained for obtaining the V. The final V soln. may be purified from Fe by evapg. to dryness again and treating as above described.

Silicate-containing materials of fat-like character and high absorptive power. ROBERT MARCUS. Ger. 322,088, Aug. 22, 1917. Sol. silicates are made to interact with hydroxy acids in suitable ratios. As hydroxy acids, lactic acid, glycolic acid or the like may be employed. The resulting salts remain emulsified in the state of finest subdivision with the colloidal silicic acid. They prevent the conversion of the silicic acid to crystalline form and are the cause of the cleansing power of the mass. The mass is used as a substitute for soap. It is claimed to be serviceable also as lubricant.

Decomposition of gang containing alkalies. FRIEDR. KRUPP A.-G., GAUSONWERK. Norw. 31,425, Nov. 29, 1920. To obtain all of the alkali and also a normal cement, the gang is decomposed by addition of CaCO_3 and the Ca salt of another acid, together with some corrigent (e. g., SiO_2), such an amt. of the Ca salt of the other acid being used as is theoretically necessary to bind the alkali by means of the acid. The gang must contain as much Fe_2O_3 as corresponds to about $1/4$ of the sesquioxides; when such is not the case, a corresponding amt. of Fe_2O_3 must be added.

Driving out potash from gang. TINFOS JERNVERK, A. S. Norw. 31,407, Nov. 22, 1920. The potash-contg. gang is fused with a blast-furnace slag which should preferably be basic and contain as much S and P as possible.

Chrome alum. G. H. HULTMAN. Brit. 159,460, Feb. 23, 1921. Addition to 138,504

(C. A. 14, 1875). According to the principal patent ferrochrome is dissolved in H_2SO_4 , and a sol. K salt is added after the removal of some of the Fe, as ferrous sulfate, by crystn. The present modification consists in the use of any sol. alkali salt, *e. g.* of NH_4 or Na, in place of K. Cf. C. A. 14, 2974.

Calcium carbide. L. P. BASSGT. Brit. 159,201, Feb. 19, 1921. Addition to 140,096. The general process for the reduction of oxygenated compds. to form metals, metal-loids, carbides, etc., by means of coal or hydrocarbon oils described in the principal patent is utilized in the prepn. of CaC_2 and the process is rendered continuous by employing a rotary furnace in the following manner. Lime or limestone and the necessary amt. of coal and fluxes are charged by means of a feeding device into the upper end of a horizontal or, preferably, slightly inclined rotary furnace and strongly heated air together with finely pulverized coal or atomized hydrocarbon oil are injected into the lower end. The quantity of air admitted is adjusted so that there is only sufficient for CO to be formed by the combustion. The combustible gases leaving the furnace are employed to heat the air before it is injected into the furnace. The general process described in the principal patent may be conducted in a rotary furnace as described above.

Catalysts for thermo-chemical reactions, more particularly for the formation of nitrogen compounds. NORL. LECHESE. Ger. 322,843, Sep. 10, 1913. Bauxite is subjected to a heating process, which consists in burning in its pores a gaseous combustible mixt. supplied under high pressure, and in taking advantage of the tendency of the bauxite to condense gases as soon as it begins to give off its hydrate water. It is claimed that by this method of heating, the bauxite is completely dehydrated in a few minutes while its chief constituents are firmly agglomerated, and its pores contracted without being stopped up. After heating, the material is slowly cooled by gradually cutting off the supply of combustible gases, while secondary air under pressure is continually supplied, nitric oxide forming until the mass has entirely cooled. Instead of bauxite, a mixt. of bauxite and carbon may be used and only air under pressure admitted, the combustible gases being supplied by the C. The ability of the bauxite to sep. or combine gases is regulated by modifying the pressure of the combustible gas mixture supplied. It is claimed that with the bauxite treated in this manner, most of the thermochemical reactions can be conducted for which Pt or like substances or different reduced metals are generally employed. For the production of nitride, charges of a mixture of bauxite and coal agglomerated to form briquets or balls are preferably employed. The atm. is kept practically neutral, but always reducing in all parts of the furnace.

Sulfur dioxide. P. PASCAL. Brit. 159,337, Dec. 2, 1919. SO_2 is obtained from dil. gas mixts., for instance gases from the roasting of blend and pyrites, by treating the gas mixt. under pressure with a carbolic oil obtained by distg. coal tar at $175-225^\circ$, and sepg. the dissolved gas by heat and reduced pressure. Then the SO_2 is liquefied. A suitable app. is specified.

Purifying zinc-containing solutions. ANACONDA COPPER MINING COMPANY. Norw. 31,336, Nov. 1, 1920. The purification is effected by the action of electrolytically pptd. spongy Zn. Cf. C. A. 14, 1294.

Gels; absorbents for gases; catalytic agents. W. A. PATRICK. Brit. 159,508, Feb. 26, 1921. Gels contg. SiO_2 or a similar acid radical and one or more metal oxides which resemble the SiO_2 gel described in 136,543 (C. A. 14, 1195), are obtained by reacting upon Na silicate, aluminate, stannate, titanate, tungstate, zirconate, or the like with one or more metal salts such as $FeCl_3$, $AlCl_3$, or $CuCl_2$, with or without a portion of free acid such as HCl. The procedure is similar to that described in the above mentioned patent. The products are suitable for absorbing gases, or for catalysts. The product obtained from $FeCl_3$ and Na_2SiO_3 is suitable for the catalytic production of sulfur trioxide. The metal/oxide may be reduced to metal. The products mentioned contain SiO_2 .

with Fe, Cu, or Ni; SiO_2 and Fe_2O_3 with Cu, Mg, Cd, Zn, or Mn oxide; and SiO_2 and Al_2O_3 with CuO .

Coated metal sheets, etc. H. H. ROBERTSON Co. Brit. 159,863, Jan. 4, 1921. A metal article such as a sheet is protected against weather and fumes by coating it on both faces and all edges with a primary coat of asphalt of low m.p. and a second coat of asphalt of high m.p. An outside film of anti-sticky material, composed *e. g.*, of viscose and a sol. soapy material, may be added.

Coated metal sheets, etc. H. H. ROBERTSON Co. Brit. 159,864, Jan. 4, 1921. Metal articles such as sheets which are coated with asphalt are rendered less sticky when packed by coating them with an aq. soln. of cellulose and soap. The cellulose is preferably viscose or viscid. The asphalted sheets may be of the kind described in 106,907 (C. A. 11, 2660) comprizing a primary layer of asphalt of high m. p., an intermediate layer of asphalted asbestos or felt, and an outer layer of asphalt adhesive only when heated.

Lampblack. FRANZ KARL MEISER. Ger. 322,612, Nov. 10, 1918. To obviate the sepn. of H_2O and the production of damp soot in the process described in 314,857, the temp. of the conveyor (soot worm) is kept, according to the present invention, above the dew point of the smoke gases. To that end the soot worm is insulated, either by insulating material on the outside, or preferably by lining the inside with insulating bricks. The circulation of the smoke gases between the chambers and the conveyor may also be prevented by shutoff devices, which may consist of automatically operating clack valves.

Phenolaldehyde condensation products. VICKERS, LTD., LOCO RUBBER & WATER-PROOFING CO., LTD., and W. H. NUTTALL. Brit. 158,447, Dec. 15, 1919. Hydrazine is employed as condensing agent in the production of condensation products from phenols (*e. g.*, phenol or cresol) and aldehydes (*e. g.*, HCHO or paraformaldehyde); colorless products are thereby obtained; the process may be stopped at the stage when resins are produced or continued to give insol. infusible products. According to the provisional specification, quinoline or piperidine may be employed as the condensing agents.

19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. R. BARTON, C. H. KERR

The industry of silica glass. G. FLUSIN. *Chémie & industrie* 5, 119-35 (1921); cf. C. A. 15, 1607.—The manuf. of the opaque and semi-translucent varieties of quartz glass is discussed. By means of the heat developed by passing a heavy current through a graphite rod embedded in pure quartz sand in a furnace, a cylinder of semi-fused quartz is formed around the resistor. The temp. is raised to such a degree that the gases formed by interaction between the quartz and the graphite core sep. the tube of quartz from the core. The tube is then withdrawn from the furnace, freed from adhering sand, and formed by drawing, blowing in molds, pressing, etc., the operations being fairly easily performed once the initial resistance due to surface cooling has been overcome. The outer surfaces of the objects are sand blasted to remove incompletely fused grains of sand. Semi-translucent ware is produced by severe sand blasting followed by treatment with HF , by forming the quartz cylinder inside a graphite cylinder so that the entire charge of sand is used and both surfaces are subjected to radiant heat, or better by reheating to the softening point. This last method results in a marked decrease in size of the bubbles which are largely responsible for the opacity.

J. B. LAIRD

The industry of silica glass. GEORGES FLUSIN. *Chimie & industrie* 5, 257-70 (1921). Cf. preceding abstract.—The manuf. of transparent quartz is described. The raw material for this variety must be of the highest purity both to avoid the discoloration caused by foreign oxides and to prevent the evolution of bubbles from chemical action. Zirconium and silicon carbide in one of their several forms are the most satisfactory refractories to date. A number of ingenious methods for avoiding or getting rid of the air bubbles occluded in the melt are briefly mentioned and patent references given. The quantity of air entrapped in the quartz mass is greatly increased by an expansion in vol. of about 1% at a temp. of 575° caused by a change in the cryst. structure to the β -quartz allotrope. Devitrification is a source of trouble and is reduced by complete freedom from bubbles and other impurities. A bibliography of 117 items is attached to the article.

J. B. PATCH

Quartz glass and the mercury arc lamp. G. BERLEMONT. *Bull. soc. encour. ind. nat.* 133, 254-60 (1921); 2 figs.—During the war B. was induced to resume his attempts to perfect the manuf. of the mercury vapor lamp. In collaboration with Henri George and others he succeeded in making a lamp for medical and experimental uses. Tungsten was employed for sealing with the quartz glass and the arc was not formed in a vacuum but at atmospheric pressure, thereby attaining two improvements: making the lamp more robust and simplifying the lighting procedure.

J. B. PATCH

The pot and block room. P. K. EDUARD SCHNURPFEL. *Glas-Industrie* 32, 169-70, 181-4 (1921).—The manuf. of pots, boots, rings and tank blocks is discussed in considerable detail. Dimensions and 8 recipes are given. The casting of pots is briefly mentioned.

J. B. PATCH

Ten years of glass machinery manufacture. ANON. *Glassworker* 40, No. 32 (1921); illus.—Historical account of the machinery of Wm. J. Miller of Swissvale, Pa.

J. B. PATCH

Meissen porcelain for pottery and chemical ware. W. FUNK. *Z. angew. chem.* 34, Aufsatzteil 127-8 (1921).—Klein (cf. C. A. 11, 287) concluded from a microscopic study that Royal Meissen porcelain was burned at a comparatively low temp. or heat treatment. Funk claims this to be erroneous.

C. H. KERR

Enamels for sheet iron and steel. J. B. SHAW. *Bur. Standards, Tech. Papers* No. 165, 88p. (1920).—A full description of enameling practice is given. Expts. were made with various American clays as substitutes for German Vallender clay used in floating the enamel frit. The more rapid the increase in viscosity with increase in specific gravity of the slip, the better the action of the clay as a floating agent. *Tennessee ball clay* appeared superior to the German clay when flocculated with $MgSO_4$, while a mixture of *Tennessee* and *Florida clays* and *Kentucky ball clay* was very good. When borax was used as the flocculant, as in ground coat enamels, the American clays were distinctly inferior to the German clay.

J. S. LAIRD

American enterprise makes "rare earth" a misnomer (PRITCHARD) 18. Preparation of zirconia from Brazilian ore and a new method of determination (ROSSITER, SANDERS) 18. The colorimetric determination of iron in silicates (MATHJKA) 7.

RICHARDSON, CHARLES H.: *Glass Sands of Kentucky*. Kentucky Geological Survey, Series 6, Vol. 1, Frankfort, Ky. 145 pp. For review see *Eng. Mining J.* 111, 830 (1921).

SEARLE, ALFRED B.: *The Clayworker's Handbook*. A manual for all engaged in the manufacture of articles from clay. 3rd Ed. revized, enlarged and largely rewritten. Philadelphia: J. B. Lippincott Co. 381 pp.

Sprechsaal-Kalender für die Keramischen, Glas- und verwandten Industrien. 1921.

Edited by J. KOERNER. Coburg: Müller & Schmidt. M 7.50. For review see *Totind.-Ztg.* 45, 464 (1921).

Light filters. E. L. FRIEDMANN & Co. Brit. 159,179, Feb. 4, 1921. Filters for lamp-shades, etc., for producing artificial daylight by gas-filled metallic filament lamps consist of glass in which cobalt-blue and nickel-green colors are melted together in one charge. Other additions such as CuSO_4 may be used for the production of a green color.

Refractory articles. O. REBUFFAT. Brit. 159,865, Jan. 5, 1921. Bricks and other refractory articles composed chiefly of SiO_2 are made from a mixt. contg. a small proportion, e. g., 0.45% of phosphoric, tungstic, molybdic, boric or other acid stable at high temps., or a salt of such acid. The bricks are baked at a temp. of 1,300–1,500° to convert the SiO_2 into modifications of low sp. gr., such as tridymite.

Refractory substances. OSMOSIS Co., Ltd., D. NORTHEALL-LAURIE and W. R. ORMANDY. Brit. 159,537, Jan. 22, 1920. Refractory articles are made by firing specially purified china clay without a flux at temps. below 1500°. The clay is purified by the methods set forth in 2379, 1911 (C. A. 6, 1964), 3364, 1911 (C. A. 6, 2036), 27,930, 1911 (C. A. 7, 1846), 27,931, 1911 (C. A. 7, 1846), 28,185, 1911 (C. A. 7, 1846), and 14,235, 1912 (C. A. 7, 4055), e. g., it is made into a slip with a dil. electrolyte such as NaOH or Na_2SiO_3 , allowed to stand for about 24 hrs., and is either subjected to electroosmosis, or pptd. by a coagulating agent such as $\text{Al}_2(\text{SO}_4)_3$, and reconverted to the sol. condition by treatment with an alkali. The purified clay is made into a slip to which a grog made of vitrified china clay may be added to minimize contraction on firing.

20—CEMENT AND OTHER BUILDING MATERIALS

C. N. WILEY

Ferroportland-pozzuolana mixtures. FABIO FERRARI. *Giorn. chim. ind. applicata* 2, 549–54 (1920).—F. comes to the following conclusions from his expts.: (1) Normal agglomerates made up of portland-pozzuolana mixts. show, in general, an incomparably greater resistance to the action of sulfates and chlorides than pure portland agglomerates, owing to the lower sp. permeability of the 1st type and its lower content in Ca aluminates. They become, therefore, passive after a longer or shorter period of time to the disintegration to which all mortars are subjected that contain strongly basic aluminates, as to alterations in their superficial continuity (the essential coeff. of mechanical resistance of every cementitious mass constituted of hydrated Ca silicates). (2) Normal agglomerates constituted of ferroportland (or portland free of binary Fe and Al compds.), in that they are free from defects derived from the presence of aluminates, undergo during long periods of time a regression because of the diffusion of CaO due to their high sp. permeability and the instability of their protective crust, characters with which they are endowed because of the high basicity of ferroportland. (3) Mortars constituted of ferroportland-pozzuolana mixts., because of the absence of both the defects mentioned above, present an abs. stability of their protective stratum and a practically perfect impermeability. Such mixts. are, therefore, the only ones rationally applicable at present to maritime construction or works intended to come into contact with sulfated waters.

ROBERT S. FOSMONTIER

Influence of additions of calcium sulfate or chloride on the shrinkage of cement. A. GUTTMANN. *Zement* 9, 310–3, 429–32 (1920).—Addition of raw gypsum or CaCl_2 to portland, iron-portland, or blast-furnace cements causes them to swell so that the natural shrinkage is counteracted or only takes place after a long time. Cements so treated are especially adapted for use as mortars where it is desired that the joint should be nearly invisible.

J. S. C. I.

Extraction of calcium oxide from calcined magnesite. L. H. DUSCHAK. *Chem. Met. Eng.* 23, 628(1920).—CaO may be almost completely extd. from magnesite after calcining at 900°–50° by leaching for several hrs. with water free from CO₂. If the magnesite has been calcined at higher temps., and especially if it is "dead burnt," the extn. is very incomplete and is not improved by fine grinding probably owing to the formation of an insol. compd. J. S. C. I.

Hydrated lime, a chemical engineering product. L. E. ALLEN. *Can. Chem. Met.* 5, 99–101 (1921).—A. reviews the historical development of the lime industry and shows the relation of the chem. engineer to its perfection. Limes are classified as: high Ca, 90% or over CaO; Ca, 85–90% CaO; magnesian, 10–25% MgO; and high magnesian not less than 25% MgO. Crushing, hydration, screening and pulverizing, packing and storing of hydrated lime are discussed and precautions given. Mechanical uses include waterproofing cement work and concrete highway construction, and results are given to show the advantages. Important chem. uses are listed. It is a stable product, not slacking, and is put up in convenient sized packages. W. H. BOYNTON

Specifications for paper makers' quicklime and hydrated lime (ANON.) 23. Decomposition of gang containing alkalis (Norw. pat. 31,425) 18.

Englische Normen für Portland-Zement. Berlin: Verlag Tonind.-Ztg. 23 pp. M. 5. For review see *Tonind.-Ztg.* 45, 481(1921).

HANISCH, AUGUST: *Zehn Jahre Portlandzement Prüfung.* Leipzig: Arthur Felix. 31 pp. M. 5. For review see *Z. angew. Chem.* 34, Aufsatzteil 159(1921).

Burning lime. N. WOLLAN and KARL SANDE. Norw. 30,821, June 14, 1920. Finely divided limestone is made to fall freely through gas heated to a high temp.

Liquid-resisting compositions for concrete. C. H. IVINSON and G. S. ROBERTS. Brit. 159,542, Oct. 28, 1919. A compn. for lining concrete tanks comprises chlorides and oxides of Mg, Zn, or both, together with NH₄Cl and finely divided Fe. One or more hardening agents such as powdered glass, Na₂B₄O₇, SiO₂, and Zn dust may be added. The Fe may be mixed with the oxides and hardening agents before the chlorides are added, or it may be added to the finished compn. In an example, 25 parts of ZnO are mixed with 1 part SiO₂, 0.5 part Na₂B₄O₇, and 0.75 part glass; to 10 parts of this mixt. are added 5 parts Fe, 14 parts MgCl₂ and 1 of NH₄Cl.

Preserving wood. H. LAUBB. Brit. 159,479, Feb. 25, 1921. To preserve wood, incisions are made in it with an instrument resembling a surgical cannula through which a preservative compd. is introduced under pressure. The preservative compd. consists of a soln. of high osmotic pressure, such as ZnCl₂, MgCl₂, CaCl₂, or sugar soln., a powerful antiseptic, such as corrosive sublimate, phenol, cresol, or compds. of Cr or fluorine, and an antiseptic which is not easily dissolved, such as powdered Cu. A neutral powder such as brick powder, powdered glass, ground brimstone or infusorial earth may be added to give the substance a pasty consistency and prevent it from escaping from the incisions.

21—FUELS, GAS, TAR AND COKE

A. C. FRIEDNER

John Downer Pennock. E. L. PIERCE. *J. Ind. Eng. Chem.* 13, 489(1921).—An obituary. E. J. C.

Certain chemical aspects of the South Wales coals; and coal field. S. R. ILLING-

WORTH. Joint Meeting of Fuel Economy Committee of Brit. Assoc. and S. Wales Inst. Eng., Aug. 26, 1920. [Advance copy.] 25 pp.—The temps. at which active decompn. commenced when typical South Wales coals were heated were found to increase with the increase of the C-H ratio in the coals. The behavior of a coal below 500°, it is suggested detcs. its characteristic properties and economic uses. The different species of coal are differentiated by the amts. of pyridine-sol. constituents they contain, the thermal stability of these substances, their stability relative to one another, and their nature. From the results it is concluded that it should be possible to produce from highly bituminous coals any desired type of semi-bituminous, dry steam, or anthracite coal, by a process of fractional decompn. It seems probable that Seyler's classification (*Proc. S. Wales Inst. Eng.* 21, no. 8 and 22, no. 3) based on the H content is on a true scientific basis, reflecting the chief substances detg. the characteristics of a coal.

J. S. C. I.

Hoo canal. Strata associated with coal. I. F. S. SENNATT and M. BARASH. Lancashire and Cheshire Coal Res. Assoc., *Bull.* 6, 15 pp. (1920).—The term Hoo canal is largely used in Lancashire for canals contg. a high percentage of ash to distinguish them from low-grade shales. Seams varying in thickness from a few inches to several feet occur in the Lancashire coal field. The av. compn. of the specimen (from Hindley) examd. was: moisture 2.10%, volatile org. matter 31.60%, coke 66.30%, fixed C 39.78%, ash 24.42%, C 60.74%, H 4.23%, N 1.30%, S 1.23%, and O 8.08%. The calorific value was 11,000 B. t. u. per lb. (14,700 B. t. u. on an ash-free basis). Ext. with pyridine yielded 12.7% of ext. compared with 29.4% of ext. from the coal associated with the canal (both figures calcd. to an ash-free basis). The ash would seem to be an inherent constituent of the canal substance, since no improvement in compn. could be effected by crushing and washing the material. Hoo canal can be pulverized approx. much more easily than Arley coal, and might be suitable for powdered fuel firing. Similar material from the Burnley area averaged 45.5% of ash and 19.0% of volatile org. matter; from the Bickershaw area 32.4% of ash and 28.3% of volatile org. matter; and from the Garswood area 10.1% of ash and 48.1% of volatile org. matter. Carbonization of the Hindley canal at 920° yielded 9870 cu. ft. of gas per ton or corrected to 16 c. p. 9210 cu. ft. at normal temp. and pressure. The c. p. was 15.0 and the calorific value 521 B. t. u. per cu. ft. The coke yield was 68.9% and the tar yield 8.0 gals. per ton (4.1% by wt.). The tar had sp. gr. 1.149 at 20° and yielded the following fractions: Up to 170°, 6.8%; 170–230°, 15.5%; 230–70°, 8.95%; 270–330°, 22.9% and 45.0% of a brittle pitch. There is a much smaller yield of naphthalene and a much larger yield of anthracene from canal tar than from coal tar. J. S. C. I.

Briqueting of lignite. LANDSBERG. *Z. Ver. deut. Ing.* 65, 415–17 (1921).—From an economic standpoint, the briqueting of lignite is both profitable and practical. In this way lignite of high moisture content and powdery nature can be utilized and converted into an asset. It makes a better fuel than raw coal, has a higher calorific value and is easier to transport. The effect of its water content upon the heat balance is discussed.

J. L. WILLY

Analysis of coal. HECTOR POULEUR. *Revue universelle des Mines; Industrie; Mat. italiana* 13, 4–5 (1921).—Review bearing especially upon the sepn. of proximate constituents of coal by various solvents and by distn. ROBERT S. POMEROY

A new method of coal analysis. H. GRÖPPEL. *Dingler's Polytech. J.* 1919, 134; *Listy Cukrovní* 38, 78 (1919).—The weighed sample of coal is placed in a glass tube, open at both ends, which is connected with another weighed glass tube filled with CaCl₂. H₂ is then passed through the app. and the system immersed in a H₂SO₄ bath, where it is slowly heated to 105°. Thirty min. is sufficient time to dry the sample completely. The increased wt. of the CaCl₂ indicates the moisture content. Volatile material is then removed by heating the dry sample in the presence of H₂. The ashing of the re-

maining coke is completed in a current of O_2 . The amt. of tar and water produced by decompn. can be detd. by condensing in a special spiral glass tube, filled with glass beads, which is attached to the app.

JOHN M. KRNO

Graphic representation of the analysis of flue gases derived from the combustion of coal. F. SCHULTE. *Glückauf* 56, 532-6(1920).—The system of triangular coordinates introduced by Ostwald (*C. A.* 13, 3305), whereby the characteristics of combustion are represented by points associated with a right-angled triangle is used to represent the results of combustion of coke, anthracite, lean coal, fat coal, gas coal, and coal very rich in volatile combustible matter. Analyses of typical samples of these various coals are given and are accompanied by triangular diagrams appropriate to the respective cases. The method of construction of the diagrams is explained, and data are provided for their construction in the case of any of the typical coals enumerated. The application of the diagrams to the detn. of the characteristics of the combustion, utilizing the results afforded by analysis of the flue gases, is briefly explained. The correctness or otherwise of the analysis is thus readily ascertained; likewise the completeness or otherwise of the combustion and whether excess of air is being used or not. J. S. C. I.

Testing casinghead gas. F. I. THOMPSON. *Oil and Gas J.* 19, No. 50, 50(1921).—A field app. more closely resembling the manufacturing process than does the ordinary single-stage gas-testing equipment is described. Instead of the single-stage high-pressure app., a two-stage vacuum pump is used. The procedure followed is, however, the same in both cases and is governed by regulations of the U. S. Dept. of the Interior. A description of the test is given. The advantage of the vacuum pump is that it allows connection with the lines without closing any gates and consequent loss of gas to the plant for several hrs.

R. L. SIBLEY

Testing generator gas. ANON. *Chem. Ztg.* 45, 365(1921).—A description of a simple app. for drawing samples for analysis from an aspirator.

J. H. MOORE

Determination of benzene hydrocarbons in illuminating and coke-oven gas. E. BEGL, KARL ANDREWS AND WILHELM MÜLLER. *Tech. Hochschule, Darmstadt. Z. angew. Chem.* 34, Aufsatzteil 125-7(1921).—The gas is passed at the rate of 250 l. per hr. through 30-40 g. dry, active charcoal (blood or sulfite waste charcoal, or that made by the $ZnCl_2$ process) in a U-tube with glass stopcocks until the charcoal has gained 25% or less in wt. The tube is then heated to 110-120° in a bath, steam is passed through and the oil condensed measured in a buret. The tube is then evacuated at the same temp.; when the charcoal is dry it is ready for another detn. As the charcoal absorbs the non-condensable vapors the gain in wt. cannot be used directly. A stripped gas gave by this method 20.2-23.9 cc. oil per cu. m.; by the dinitrobenzene method 16-17.2 cc.; by the paraffin stripping method 9.74-10.1 cc. Deville's freezing method could not be used at all.

ERNEST W. THIGLE

New gas calorimeter. E. LANGTHALER. *Gas und Wasserfach* 64, 83-6(1921); 2 figs. —The Union Calorimeter designed by O. Dommier is described. The principle is similar to that of the Strache calorimeter (*C. A.* 4, 1826) except that the heat of explosion of the gas is taken up and measured by the expansion of petroleum oil instead of that of air. Also the standard H gas is made within the explosion pipet itself by electrolysis of a dil. soln. of H_2SO_4 instead of being drawn in from an outside source. Small amts. of gas can be measured irrespective of the gas temp. or pressure or the barometer pressure. Its detns. for gases from 500 to 8000 cal. agree very closely with detns. by the Junker calorimeter. Its operation is simple and efficient.

J. L. WILBY

Long-distance pressure indicator. H. C. WIDLAK. *Gas J.* 154, 376-8(1921); 5 figs. —A device is described for giving at a gas-works a continuous visual or graphic record of all pressure variations within the outlying supply mains. The app. is a circular water-filled tank so constructed as to form a U tube, the outer limb being connected to the gas system and receiving the pressure, the other carrying a mercury-filled float which

risers and falls with changes in the water level induced by variations in the gas pressure, and makes elec. contact with a number of resistance elements. Thus any variation in the pressure of gas supplied to the gage effects a corresponding alteration in the amperage flowing in the elec. circuit in which it is connected. In this manner the amt. of current flowing in the circuit at any moment is proportional to the pressure of gas within the body of the gage, and being transmitted to the works for measurement, is read on the dial of a meter calibrated in tenths of an inch of gas pressure. This app. is already in use and operates over a long period of time with only slight attention to cleaning the contact needles once or twice a month. .

J. L. WILBY

Water gas apparatus and use of Central District coal as generator fuel. WM. W. ODELL. *Bur. Mines, Tech. Paper No. 246*, 28 pp.(1921); cf. *C. A.* 15, 1204.—O. describes some of the modern types of water-gas app. such as the Loomis, Rew, Kramers and Aarts and the Smith modification, discusses heat balance and blasting reactions and compares the use of coke and coal. In designing a generator set best suited for Central District coal there are certain operating possibilities to be considered: (1) shorter operating cycles are not recommended; (2) a combined retort and generator is practicable so that the coal can be partly coked before it enters the generator; (3) admission of air in small amts. with the down-run steam offers a means of increasing the production of blue gas; (4) use of highly superheated steam is desirable; (5) the steam should be admitted below the coking line in the generator to avoid retardation of the coking of the green coal; (6-7) using a deeper fuel bed and utilizing the excess generator blast gas in waste-heat boilers thus allowing the use of proportionately larger generators than the checker chambers accompanying them; (8) blowing secondary air into the generator just below the coking line thus reducing the % of combustible gas in the blast gas and avoiding the excess of heat from this source; (9) employing heated air as generator blast thus transmitting to the green coal more heat and producing higher temps. in the generator. There is danger, however, of burning out the grates; (10) elimination of grate bars and slagging of clinkers by means of hot air blasts in a generator built along the lines of a blast furnace. This will eliminate the long clinkering periods and increase the efficiency. A desired temp. in the fuel bed is 1300-1400°. With a rate of air blast per min. per sq. ft. of grate area of 160 cu. ft. and a fuel depth of 7.5 ft., 3 sec. is a practical time of contact. At 1200° the blast gas will contain approx. 18% of CO. In the water gas reaction it is desirable to have the fuel bed as deep and the temp. as high as possible and a long time of contact is desirable in order to decompose the greatest amt. of steam with formation of a minimum amt. of CO₂. At 1300° the time of contact of the steam and fuel should not be less than 2 to 4 sec. in order to have the minimum amts. of water vapor and CO₂ in the gas.

J. L. WILBY

Water gas industry. J. SCHMIDT. *Z. Ver. deut. Ing.* 65, 418-21(1921).—S. reviews the use of water gas as the heating medium in the iron and metal industry for melting, tempering, welding, etc.

J. L. WILBY

Complete fuel service by utilities. C. R. BELLAMY. *Gas Age* 47, 416-20(1921).—B. discusses the uses and advantages of coke-oven gas made in Semet-Solvay ovens for use as city gas. By using blue gas for oven fuel, from 60 to 100% of the total rich coke-oven gas can be made available for city consumption. In extreme cases, this coal gas can be supplemented by carbureted water gas. A good margin of profit can be assured to the gas company installing a coke oven plant for its gas supply. B. does not favor complete gasification of the residual coke; the mixed gas being of too low grade for domestic use, and besides the coke can be more economically and efficiently used as a solid fuel.

J. L. WILBY

Use of exhaust steam in water gas manufacture. H. KEMPKERS. *Hot Gas* 1920, 172-3; *Gas und Wasserfach* 64, 192(1921).—At the Oofter gas works, Amsterdam, waste steam is collected from the various operations, freed of its oil by impact, and

preheated by the hot water gas from 98° to 130°, and then passed through a second preheater where it is heated by the blast gases to 250° to 300°. It is then utilized in the gas producer. The advantages of the method are: the extreme heat saving, the regular production of gas, shortening the blast period from 3 to 2.5 mins., and increasing the gasification period from 5 to 7.5 mins., increased production of gas from 140 to 160 cu. m. to 200, saving of fuel in the boiler house, and the decreased amt. of unchanged water vapor in the water gas. The process is especially applicable to small and medium-sized plants.

J. L. WILEY

Reconstruction work at the Newark gas works. F. A. WEST. *Gas J.* 154, 259-63 (1921).—Among the improvements, a *Tully carbureted hydrogen plant* was installed and the 330 B. t. u. gas mixed with the coal gas from the retorts by passing it directly into the coal-gas foul main. The make of mixed gas per ton of coal was 16848 cu. ft. of 426 B. t. u. gross: coke sold 7.5 cwt., tar 10.6 gal. and NH_3 liquor 42.8 gal. Considerable tar is recovered also from the Tully plant, and the crude carbureted hydrogen gas contains 76 grains of NH_3 per 100 cu. ft. and 13.5 grains of S compds. but no CN. The H_2S + other S compds. in the coal gas averages about 520 and the HCN 127 grains in the coal gas and 50 grains in the mixed gas before entering the purifiers which remove all traces of that as well as 6 grains of SO_2 . CO content averages about 15-16% in the mixed gas. The carbureted hydrogen gas analyzes when making 65000 cu. ft. per ton, CO_2 4.9, O 0.4, CO 28.4, C_mH_m 1.3, CH_4 5.55, H 50.7 and N 8.75%. The inerts are 14%. The cost of the mixed gas to the holders was 2s. 6.2d. per 1000 cu. ft.

J. L. WILEY

Gas and its by-products. A. GREDEL. *J. usines gaz* 45, 97-101, 114-9, 130-2 (1921).

—A review of development and methods.

J. L. WILEY

An American gas purification investigation. W. A. DUNKLEY and C. E. BARNES. *Gas J.* 154, 27-9, 148-50, 204-5, 266-8, 381-2 (1921); cf. *C. A.* 14, 2255.

J. L. WILEY

Principles of the Tully gas plant and its adaptation to modern gas works practice. C. H. CHRISTER. *Gas J.* 154, 323-6 (1921); *Gas World* 74, 384-7 (1921).—A Tully plant consists essentially of a retort superimposed on a generator. The distn. of the coal is accomplished in 2 ways; by the heat generated by the combustion of the waste gases, and by the passage through the coal charge of the hot water gas at a temp. of about 1000° generated in the producer below. It is a sort of low-temp. carbonization in a protective atm. of water gas. The temp. in the retort is about 450° at the top, gradually increasing to 1200° at the lower end. This method of carbonizing produces a rich gas at a higher efficiency than can be obtained with retorts and a sep. water-gas plant. The yield of gas may be usually taken at 60,000 cu. ft. of 340 B. t. u. or 20,400,000 B. t. u. per ton of coal. The coal gas and the Tully gas are mixed in the proportion of 45 to 55%, giving about 20,000 cu. ft. of mixed gas of 476 B. t. u., analyzing CO_2 5.8, O_2 1.0, CO 22.0, C_mH_m 0.9, CH_4 19.7, H_2 45.0, N_2 5.6, total inerts 12.4%. Yield of sale coke is 7.57 cwt. and tar 10.58 gal. Comparative costs per 1000 cu. ft., Tully gas 10.6 d., coal gas 24.08 d., and mixed gas 18.9 d., or a net saving of 5.18 d. after allowing for fuel for steam-raising. The tar has the general characteristics of low-temp. tar, the presence of paraffins being indicated. (Cf. Moore, *C. A.* 14, 333; Rule, 15, 588, 1799.)

J. L. WILEY

Complete gasification at Harrogate. F. H. ROBINSON. *Gas J.* 154, 327-9; *Gas World* 74, 368-71 (1921); *Gas Age* 47, 462-4 (1921).—Further details are given of the construction and operation of a Robinson's mixed-gas plant (cf. *C. A.* 15, 1066). The cost of manufg. gas on a 480-B. t. u. basis is 12.27 d. per 1000 cu. ft. or 2.5 d. per therm; a saving of 1 d. per therm over coal gas. In one test carbonizing in 20 hr. 2.8 tons of coal, 55000 cu. ft. of 344 B. t. u. gas, equal to 18,920,000 B. t. u. per ton of coal were obtained, or 63.1% of the heat value in the coal.

J. L. WILEY

Fractional distillation with contact-ring still-heads. R. LASSING. *Gas J.* 134, 330(1921); *Gas World* 74, No. 1920 (Coking and By-products Sec.), 17-18(1921); *J. Soc. Chem. Ind.* 40, 115-9T.—The rings described consist of cylinders of approx. equal height and diam., having a slit in the circumference and a diametrical partition connected to one side of the slit. They are placed indiscriminately in a cylindrical still-head, the resistance therein being almost negligible owing to the large amt. of free space remaining about 87%. The fractionating efficiency is very high; it is due to the large surface per unit of volume, the turbulence caused by frequent deflection of currents of vapors and condensate, and the consequent absence of channeling. Max. efficiency is obtained by lagging or heating the still-head and by superimposing a reflux condenser. The rings may be used in scrubbers, condensers, heat-exchangers, etc. To show its efficiency, from a mixt. of equal parts of benzene and toluene, 40% of the former distd. within less than 1°, and a like portion of pure toluene, leaving only about 5% of the total vol. in the mixed middle fraction. From a crude coke-oven benzene, almost all of the benzene and toluene was distd. off, each within 0.2°, though the sample contained 30% of naphthalene and wash oil.

J. L. WILBY

Rational utilization of moist bituminous lignite. T. LUNSGAARD. *Braunkohle* 19, 237-9(1920).—Distn. of moist lignite in fireclay retorts or in gas producers is disadvantageous for several reasons, and the use of zone producers, such as those described in Ger. pats. 302,322, 303,954, 313,470, and 322,648 (*J. Soc. Chem. Ind.* 37, 831A; 38, 890A; 39, 441A, 650A), is recommended. The formation of pasty nuclear layers is thereby prevented, and no decompn. of the tar takes place, as it is diluted and removed from the app. at the time of formation.

J. S. C. I.

Distillation and gasification of bituminous material. BUSK. *Braunkohle* 19, 201-6(1920).—An app. for the distn. of bituminous material designed to provide a graduated heating, to prevent local overheating, and to produce an increased yield of ammoniacal liquor, consists of a shaft furnace in which the material is heated to successively higher temps. as it passes down over a series of horizontal partitions. A rotating arm attached to a central shaft sweeps over each partition and, after a complete revolution, the material falls on to the partition below. Air is introduced at the lowest partition where the coke burns with the formation of CO₂; on the partitions above the CO₂ is reduced to CO; in the next higher zone, in which the gas is cooled by the addition of gas liquor vapor, distn. takes place; while in the uppermost zone the material is dried. Portions of the residue may be removed from any of the lower partitions, leaving only that necessary for combustion.

J. S. C. I.

Vertical chamber ovens at Eindhoven (Holland). J. ROEDERSBURG. *Hel Gas*, Nov. 1, 1920; *Gas u. Wasserfach* 64, 182-4(1921).—A block of 7 Klönne chamber ovens has been in operation since 1918. The plant and operation are described. With a low-grade coal used at first 340-370 cu. m. of straight coal gas of 4400-4800 cal. were produced per ton of coal. Its compn. CO₂ 1.6%, heavy hydrocarbons 3, O 1.9, CO 7.3, H 49.2, CH₄ 23, and N 14. Later, steaming for 3-6 hr. was practiced; the gas yield increasing to 390-440 cu. m. and the heating value decreasing to 4300-4400 cal. The mixed gas had a compn. of CO₂ 2.8%, heavy hydrocarbons 2, O 1.1, CO 12.4, H 50.9, CH₄ 19.9 and N 10.9. The coke is of metallurgical quality with only 3-7% as breeze and fine coke. From 10.57-12.5 kg. of (NH₄)₂SO₄ are produced, and 38.1-38.5 kg. of tar per ton of coal. The tar is thin with a sp. gr. of 1.11-1.14, a C content of 3%, light oil 4% and 2-4% of water. The fuel requirement for heating the ovens is 16% of the coke produced. The cost of the gas to the holder ranges from 69.14 to 70.61 florins per 1000 cu. m.

J. L. WILBY

Gas main stoppages. J. DOLLINGER. *Z. Ver. Gas.-Wasserfach* 61, 21-4(1921).—D. contradicts the statement of R. Mezger (*Ibid* 1919, 197; *Wasser u. Gas* 1920, 280) that benzene in gas does not have a protecting influence on gas piping, and offers

data to show that with washed gas stoppages increased to a considerable extent owing to naphthalene deposits and to rust particles collecting in the elbows, these stoppages being more acute in the cold months. The benzene seems to coat the inside of the pipe with a greasy, protecting film which prevents attack. The corrosion of piping when debenzolized gas is used can be prevented only by removing the water vapor, O and CN compds, as well. Galvanized pipe does not seem to offer any superiority over iron pipe against corrosion.

J. L. WILEY

Increasing the quantity and quality of tar by cooling the raw gas between the oven and the holder. BINDER. *Gas u. Wasserfach* 64, 129-30(1921); 1 fig.—Cooling of the gas is effected by spraying water into the main, washing out the tar and preventing deposition of pitch. The mixt. of tar and water is drawn off and settled. The method has resulted in the recovery of about 25% more tar, which on distn. yields about 30% more light oil, 7% more middle oil and a corresponding lesser yield of the heavy oils and pitch.

J. L. WILEY

Central generators for inclined retorts. E. KÖRTING. *Gas u. Wasserfach* 64, 190-1(1921).—The advantages of having central generators for the inclined retort plant with regeneration are: greater output of large coke, less labor requirement, saving in fuel and increase in the life of the retorts.

J. L. WILEY

Comparison of methods for producing tar and gas from lignite. H. HOLZWARTH. *Braunkohle* 19, 189-92(1920).—Four methods of treatment of lignite contg. 8% of bitumen have been compared, viz: (1) The moist lignite was directly gasified in producers; (2) the lignite in the form of briquets contg. 15% of moisture was gasified with recovery of tar and by-products; (3) partial carbonization in rotary ovens to obtain tar and burning coke; (4) partial carbonization in Thyssen rotary ovens for production of burning coke and subsequent gasification of the coke producers. The following results were obtained, calcd. to the raw, undried lignite:

	1.	2.	3.	4.
Consumption of raw lignite in production of 1 million calories in the gas.....	700 kg.	1805 kg.	925 kg.	800 kg.
Tar yield in production of 1 million calories in the gas....	28 kg.	31 kg.	29-3 kg.	63.5 kg.
Ratio raw lignite consumed: tar produced	25	35	31.6	12.5

J. S. C. I.

Calculation of the efficiency of complete gasification from the analysis of coal. H. STRACHE and A. GROSS. *Mitt. Inst. Kohlenvergasung* 2, 4-5, 45-6, 50-3, 59-61, 69-71 (1920); cf. C. A. 14, 3518.—The efficiency of the various methods of gasification were calcd. for different varieties of coal, taking as typical cases the production of semi-water gas, Mond gas, and compd. gas ("double gas" or "tri-gas" processes). The efficiency calcd. on tar-free coal varies (for the coals cited), for semi-water gas between 66.0 and 78.7%, for Mond gas between 47.2% and 52.1%, for compd. gas ("double gas") without pre-drying and with excess steam between 74.0% and 83.7%, and for compd. gas with pre-drying and without excess steam between 54.8% and 69.8%. In the semi-water gas process, by the use of warm water for moistening the air, an efficiency of 65-80% can be attained, according to the ash and water content of the coal; while in the Mond gas process the efficiency varies from 45 to 55%. The compd. gas ("double gas") process without pre-drying is only practicable if the ratio of the water content of the coal to the ash-free coke is less than 2.60. If this ratio is smaller than 0.5, 10-20% excess steam can be used. The total efficiency lies between 80% and 85%. The compd.-gas process

with pre-drying is only practicable if the ratio of water to ash-free coke is more than 2.60, or if the yield of NH_3 makes it worth while. The efficiency of this process is 70-76%. The efficiency is not, however, the only criterion of value of a process. Other factors may be of more importance, e. g., the calorific value of the gas (especially where it is necessary to attain high temps.), or the NH_3 yield, which is high in the Mond process, although the efficiency is low. The quality of the tar again varies greatly according to the process adopted. The compd.-gas process would be used where high-flame temps. are necessary, where the gases are to be conducted to long distances, or where the yield and quality of the tar are of great economic importance. J. S. C. I.

The present condition of the coal-tar industry. ERICH GLASER. *Oesterr. Chem. Ztg.* 24, 50-3, 58-61, 66-8(1921). E. H.

By-product coke plant of the Jones & Laughlin Steel Co. C. R. MEISSNER. *Chem. Met. Eng.* 24, 891-4(1921).—An illustrated description. E. H.

Development of the by-products coke oven in the United States. L. LITINSKY. *Essen. Feuerungstechnik* 9, 105-6(1921). C. J. WAST

Swelling of coal during coking. F. KORTEN. *Stahl u. Eisen* 40, 1105-8(1920).—The tendency to swell during carbonization possibly depends on the variation in temp. and mode of decomp. of the bituminous cementing material (cf. Fischer and Gluud, *C. A.* 14, 2071). K. proposes a test for the swelling on coking:—100 g. of the coal is coked under standardized conditions in a cylindrical crucible of iron closed by a perforated piston. The latter supports a weighted lever fixed at one end so that the free end moves in accordance with the rise or fall of the piston. The free end is made to trace a magnified record of the movement of the piston, i. e., of the change of vol. of the coal, upon a revolving drum covered with paper. The results agree with large-scale practice. A coal with 27% of volatile matter which showed a contraction of 4% was known to be easily coked and pushed from the oven. Another coal with 14% of volatile matter expanded nearly 6% during carbonization and 3.5% at the end. In practice the coke from this coal was difficult to push from the ovens, and then only with damage to the walls. J. S. C. I.

Regulation of gas pressure in coke-ovens. A. THAU. *Glückauf* 56, 901-6, 925-30 (1920).—No coke-oven being a gas-tight chamber, pressure differences between the chamber, the heating flues and the outside air lead to leakages. With a positive pressure in the oven the consequences are loss of gas, and C deposits in the heating flues. With a negative pressure partial combustion of the gas takes place in the oven and the resulting rise in temp. damages the exit pipes and leads to decreased yields of tar, benzene and NH_3 , and to poor gas. For ordinary working a slight positive pressure should be maintained. This principle applies to all retort practice, with or without steam, and whether gas or coke is the main desideratum. The idea that the yields of by-products can be increased by applying suction is erroneous. In many coking plants the pressure is regulated by the aid of a control flame allowed to burn on a branch pipe from the gas main. Detailed pressure measurements taken on a Koppers oven, with corresponding gas analyses, are given to show the defects of this method. The pressure is lowest at the two end doors of the oven, somewhat higher along the top, and much higher in the gas main. Hence suction on the oven and a bad gas compn. can co-exist with pressure on the gas-main and the control flame burning. The device of allowing control flames to burn at the observation holes of several oven doors is preferred. Attempts to devise an automatic control operating from a manometer to the blowers have met with great difficulty owing to the tar vapor and temperature of the gas. To render such control practicable the difference of pressure between the ovens and the point in the main connected with the manometer, which varies for each plant and may vary in the same plant with the moisture content of the coal and the deposition of C in the pipes, must

be accurately known. A description of a membrane micromanometer and of the mechanical details of automatically controlled blowers is given. J. S. C. I.

Cleaning of blast-furnace gas (FOWLES) 9. Substitution of lignite of San Giovanni Valdarno for coke with the Perrin sulfuric acid concentrating apparatus (LINARI) 18. Oil production from shale and coal (BOSTAPH) 22. The recovery of ammonia from the gases escaping during saturation (RUGGER) 28. Ammonia; salts of acids obtained from peat (Brit. pat. 159,193) 18.

CHRISTOPHER, J. E.: *Modern Coking Practice*. Including Analysis of Materials and Products, by T. H. Byrom. 3rd Ed. enlarged. 2 vols. London: Crosby Lockwood & Co. 21s. For review see *Gas World* 74, (Coking and By-product Sec.) 19(1921). Cf. C. A. 12, 1117.

ELLIS, CARLETON and MEIGS, JOSEPH V.: *Gasoline and Other Motor Fuels*. New York: D. Van Nostrand Co. 670 pp. \$10 net. For review see *Mining Sci. Press* 122, 732(1921).

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Fuel. L. W. BATES. Brit. 159, 173, Jan. 5, 1921. Fuel consisting of a mixt. of coal dust or other carbonaceous particles and liquid hydrocarbon is congealed to a paste, semi-solid or solid by cooling to at least 40° F. so as to prevent settling out of the coal to form a protection against the explosive effects of shells and torpedoes on a ship using such fuel, and to render it fireproof, especially when kept below H₂O.

Powdered fuel. N. K. H. EKLUND. Swed. 46,957, Apr. 14, 1916, and 46,958, Aug. 5, 1916. Finely ground and well dried mineral coal or charcoal is mixed with finely divided well dried pulverized peat which, before being finely ground, has been rendered non-hygroscopic by artificial drying at high temp. (e. g., 100°).

High-grade dense coal from hydrous fuel. CARBOZIT, A.-G. Norw. 31,290, Oct. 25, 1920. In order to preserve the density of the raw materials, such as lignite, peat, wood, etc., they are dried before distn. at less than 100° in moist air in counter-current until their H₂O content has been reduced to 10-20%.

Recovering coal. E. EDSEY, H. L. SULMAN and F. B. JONES. Brit. 159,285, Nov. 20, 1919. Coal is recovered from coal-bearing materials such as dross, refuse, shale, washings, etc., by froth-flotation in liquid containing in soln. a modifying or deflocculating agent, such as Na₂SiO₃, H₂SiO₃ sol., Na₂CO₃, NaOH, or other alkali, or mixts. of these. A small proportion of sol. frothing agent such as cresol may be present, with or without an insol. frothing agent, such as paraffin oil; or vegetable infusions such as saponin, cinchona or quassia may be used. The material may be ground in the presence of the modifying agent. Sea water or a soln. of an inorg. salt may be used as the pulp-forming liquor, the salt acting as the modifying agent. Cf. 122,454.

Treating alcohol for making motor fuels. CHEM. FUEL CO. OF AMERICA. Brit. 159,880, Mar. 3, 1921. Alc. is treated to remove H₂O and render it miscible with hydrocarbon oils such as petroleum oils, benzene and tar oils, to produce motor fuels, by mixing it with fusel oil and hydrocarbon oil and distg. the mixt. The first fractions, which contain H₂O, are treated to sep. alc., etc., and the final fractions which are practically free from H₂O are blended with hydrocarbon oils. In the case of low-grade alc. which contains fusel oils, a further addition of fusel oil is not necessary. A motor fuel prepd. by blending the treated alc. with petroleum oil may be mixed with ether to give quicker ignition and with toluene to prevent loss of the ether.

Briquets from coal dust and peat slime. HANS NIELSEN. Norw. 31,291, Oct. 25, 1920. Coal dust 49, peat slime 49, pitch $\frac{1}{2}$, and tar $1\frac{1}{2}\%$ are mixed by stirring, while heated to 40° , until the mass is practically free from H_2O . While hot it is briquetted at a pressure of 60 to 70 kg. per cc.

Manufacture of briquets. AKTIESELSKAB DE FORENEDE KULHANDLERES BRIKET-FABRIK. Norw. 30,746, May 25, 1920. For the manuf. of briquets in which tar is used, unslaked lime is added to the powdered fuel, and the heat liberated upon the slaking of the lime with the moisture of the fuel or of specially added H_2O used to render the tar thinly liquid, so that in the subsequent compression of the mass to briquets it will be forced in between the particles of fuel and bind them.

Briquetting coal. CHARLES HOWARD SMITH. Norw. 31,320, Nov. 1, 1920. Bituminous coal is treated in such a manner that it will give off all gases and vapors volatilizing at low temp., while at the same time it swells and becomes porous. The dist. is then discontinued, the residue pressed into briquets, after addition of a binding agent if required and such briquets are distd. at higher temp., e. g., $750-1010^\circ$, until all the gases and vapors have been driven off. The result will then be a dense connective pressed coal of a low degree of porosity.

Treating carbonaceous materials. W. E. TRENT. Brit. 159,497, Dec. 15, 1920. Addition to 151,236 (C. A. 15, 592). The agglomerated mass obtained as described in the principal patent is treated in order to recover the agglomerating agent. The agent for the agglomerating process may comprize oils extd. from coal. After being thinned with hydrocarbon liquid and commingled with air or gas, the mass is supplied from a hopper into a pipe extending through a retort, which is heated by burning a combustible mixt. delivered through a pipe. The material is carried through the retort in the current produced by a pump and the treating agent is volatilized together with the volatile constituents of the solid particles, which latter may be sepd. in a dry settling chamber. The distd. substances pass off through a pipe. Sepn. of fine ash particles contained in the carbonaceous matter is facilitated by expelling the volatile content explosively by heating with sufficient rapidity or by subsequently passing the particles through a zone of lower pressure. The volatile constituents and C particles may be delivered to a wet collecting chamber receiving H_2O from a pipe. Agitation of the condensed vapors causes the C and oils to agglomerate and exclude the non-carbonaceous particles and H_2O .

Carbonizing. M. GERCKE. Brit. 159,243, Oct. 20, 1919. In the continuous distn. of coal and shale by contact with steam superheated to about 500° , the fuel is preheated before it enters the distn. chamber or comes into contact with the steam. A suitable construction is specified.

Benzene mixture liquid at low temperature. TETRALIN, G. M. B. H. Ger. 329,833, Mar. 29, 1916. A benzene free from or poor in toluene is mixed with dekahydronaphthalene or with bydronaphthalene mixts. consisting chiefly of dekahydronaphthalene.

Gas manufacture. W. O. FELT. Brit. 159,905, Sept. 22, 1917. Coal, oil, or producer gas is purified by supplying it with glycerol, compressing it in two stages to a pressure of 300 lb. per sq. in., and passing the mixt. through a condenser maintained at about a temp. of $60^\circ F$. provided with baffles and a drain cock for the condensed glycerol and impurities, and then through a separator or extractor which completes the removal of the glycerol, etc. A suitable construction is specified.

Carbon. I. SZARVASY. Brit. 159,823, Feb. 28, 1921. Addition to 158,891 (C. A. 15, 1973). The parent specification describes a process for the production of pure retort C by the decompn. of CH_4 in the presence of pure C previously made by the same process. The present process consists in the employment of retorts constructed from such C at any rate in the tubular or other parts subjected to high temp. for the conduct of the reaction. C filling may be employed as before, and the C parts of the

retort finally may be broken up with some of the contained C for the manuf. of other retorts for the conduct of the process.

Automatic slag-removing construction for water-gas generators. E. DOLENSKY. Can. 210,552, Apr. 19, 1921.

Preheating coke-oven gases. ARTHUR ROBERTS. Can. 210,706, Apr. 19, 1921. One of the constituent gases for the burners of a coke oven is preheated by mixing with it spent gas at substantially the temp. at which it leaves the heating wall of the oven.

Preheating coke-oven gases. ARTHUR ROBERTS. Can. 210,707, Apr. 19, 1921. A coke oven has means for delivering to the burners a mixt. of a constituent gas and spent gas coming directly from the heating walls at substantially the temp. at which the spent gas leaves the heating walls so that the mixt. is preheated by the use of a minimum amt. of spent gas.

Heating coke-oven walls. ARTHUR ROBERTS. Can. 210,708, Apr. 19, 1921. Coke-oven walls are heated by passing hot gases through the body of the walls and causing the gases to impinge against portions of the wall repeatedly and with an increasing amt. of violence as the gases approach the point of exit to eliminate any tendency towards the formation of layers of inert gas in the walls.

Regenerative coke ovens. ARTHUR ROBERTS. Can. 210,709, Apr. 19, 1921. In a bench of coke ovens there are in combination with the heating walls, a common connection for each wall for the delivery of air to all portions thereof, a regulator under each end of each heating wall and connections from each of the regenerators to the common connection of the corresponding heating wall.

Regenerative coke ovens. ARTHUR ROBERTS. Can. 210,710, April 19, 1921.

Coking peat. TORFVERWERTUNGS GES. DR. POHL UND VON DEWITZ (formerly TORFVERWERTUNG DR. POHL KOMM.-GES.) Brit. 159,464, Feb. 22, 1921. Addition to 158,513. In a process of coking raw peat and the like alternately under pressure and vacuum, as described in the principal patent, the peat is compressed by providing in the container a plate whereby pressure is applied to the charge, *e. g.*, by means of a screw. This plate may serve to sep. dry, compressed peat from peat which is still being dried, the actual coking taking place on one side of the plate and the drying on the other.

22—PETROLEUM, ASPHALT AND WOOD PRODUCTS

F. M. ROGERS

Properties of typical crude oils from the producing fields of the Rocky Mountain district. E. W. DEAN, M. B. COOKE AND A. D. BAUER. Bur. Mines, *Repts. of Investigations*, No. 2235, 50 pp. (1921).—Twenty-four samples of crude oil representing the production of the district, have been analyzed by the Bureau of Mines method for the distn. analysis of crude petroleum (Dean, *C. A.* 15, 1209). Complete tables show the source of the samples, and the results of the tests. A majority of the samples are normal, *i. e.*, the properties of the successive fractions show consistent differences, indicating no change in the classes of hydrocarbons present. The crudes show a high rating on the basis of gasoline content (20% in most important from point of view of production). Some samples yield 40% of naphtha distillate. A sample from Pine Mountain field (Wyoming), had no fraction that distd. at atm. pressure to 275°, and showed an abnormal tendency to crack when heated. From the Cat Creek field (Montana), a sample was obtained that consisted almost wholly of naphtha and kerosene (90%). W. F. FARAGHER

Optical rotatory power of Roumanian petroleums. CR. PREDESCU. *Bull. soc. sci. acad. Roumaine* 6, 181-8 (1919-20).—The literature shows a number of contradictions concerning the optical rotatory power of various Roumanian petroleums. P. has ex-

aminated two samples of crude oil from Câmpina with a new Pellin polarimeter using 50-, 200-, or 500-mm. tubes. The paraffin-free sample (d. 0.8671), was fractionated into 10° fractions at atm. pressure. The fractions between 50° and 220° showed no optical activity (500-mm. tube, (Na) D light) the higher fractions showed very slight + rotation under the same conditions. Another crude from the same source (d. 0.8750) showed similar behavior. The fractions above 260° were dissolved in C_4H_{10} or in CS_2 . The refined burning oils were devoid of activity while the crude distillates showed very slight + rotation. Distn. at 15 mm. yielded fractions with the following optical properties: 90-150°, inactive (200-mm. tube); 150-180°, very slightly + (200-mm. tube); 180-200°, slightly + (200-mm. tube); 200-220°, slightly + (100-mm. tube); 220-230°, same as preceding; 230-240°, + 0.7° saccharimetric scale; 240-50°, + 1.10; 250-60°, same as preceding; 260-70°, + 3.3°; 270-80° + 3.8°, all in 100-mm. tubes, calcd. to 200-mm. tube. The fractions above 250° were dissolved in C_4H_{10} or CS_2 , and the rotations calcd. by the standard formula. The residuum (above 350°) was so highly colored, that 0.002 g. dissolved in 10 cc. of solvent allowed scarcely any light to pass. No difference in the two fields was observable with these samples. The crude oils were examd. in the same way as the residuum. (1 vol. to 240 vol. of C_4H_{10} .) There was no evidence of optical activity. In CS_2 , a slight + rotation was observed with 100-mm. tube. The same uncertain results were obtained by dissolving the crude oil in its own naphtha fraction. Residuum and crude oils were filtered through fuller's earth (downward through 6 cm. of earth under the pressure of a 15-cm. column). A slightly reddish yellow oil was obtained from the more dense crude. A reddish filtrate was obtained from the other crude oil. The optical activity was as follows: $[\alpha]_D^{20} = +1.19$ for the first and +1.52 for the second crude oil. The solvents alone when passed through fuller's earth showed no optical activity. Direct examn. of layers of crude oil 1 mm. thick was impossible, since no light passed through (electric arc). Two light colored crude oils (Govora and Pârjol-Câmpeni) as well as white naphtha from the Caucasus, Szurachany, Velleja and Montechino were found to be optically inactive.

W. F. FARAGHER

Capillary constants of Roumanian petroleum. CR. PREDESCU. *Bull. sec. sci. acad. Roumaine* 6, 188-96(1919-20).—The capillary const. (Laplace formula) of the fractions (to 270°) of a crude oil from Câmpina (d. 0.875) increased gradually from 2.20 to 2.89 (15°). The crude petroleum had a capillary const. of 2.77. The viscosities of these fractions in Engler degrees, increased from 0.910 to 3.131. The capillary const. shows greater variation for these fractions than do the densities and the indexes of refraction, which are more frequently used in analytical practice. The viscosity of the fractions increases much more rapidly than the capillarity. In this fact is found the reason why fractions with high b. p. rise more slowly into a capillary than the fractions with low b. p. (Rate of rise is inversely proportioned to the viscosity.) The united action of the two properties, capillarity and viscosity, explains the law of Lisenko and the phenomenon of adsorption by capillary diffusion; the lighter fractions are able to pass through the porous layer the more rapidly. In addition, the phenomenon of adsorption must be considered. The capillary const. were found to decrease slightly (about 1%) with time (16 months), while the Engler degrees of the samples increased during the same time (5%-15%).

W. F. FARAGHER

Burning petroleum. E. C. BOWDEN-SMITH. *Pet. Times* 5, 581-2(1921).—This article is a discussion of the various processes of using petroleum products in which combustion takes place. Burning of crude petroleum is a wasteful practice. Although the internal-combustion engine is highly efficient, a great amt. of heat is wasted. The full heating power of petroleum is best utilized when it is burned under boilers, where it can maintain a uniform output of steam from the generator and nearly 100% greater vol. or higher evapn. than with solid fuel.

R. L. SMITH

Use of alcohol in refining petroleum distillates. Z. BUDREWICZ. *Przemysł Chem.* 4, 63-4(1920).—In the refining of Baku solar oil with H_2SO_4 , alc. is added in some factories to remove the sulfonic acids, aq. solns. of which form persistent emulsions with mineral oils. The oil, after treatment with H_2SO_4 , is warmed to 50° , treated with 0.25-0.3% of alc., shaken for 1 hr., and allowed to stand. The sulfonic acids are then run off and the oil is washed. In this way 7% of sulfonic acids have been produced from Baku solar oil. They possess detergent and emulsifying properties in acid, neutral and alk. solns. and in hard waters. J. S. C. I.

New technical methods for separating aqueous oil emulsions. I. MÓSCICKI AND K. KLING. *Przemysł Chem.* 4, 2-8(1920).—In accordance with the method previously suggested (*C. A.* 14, 833), aq. mineral oil emulsions may be sepd. by maintaining them for 2-4 hrs. at $120-40^\circ$ under 2-6 atm. pressure in a perfectly air-tight pressure vessel. The emulsion may be forced continuously under pressure through a preheating system into a vertical holder, in which it slowly rises. The dimensions of the holder are such that the emulsion remains in it for several hrs. at a temp. high enough to effect sepn. The sepd. oil is discharged through a pressure valve at the top of the holder, and the sepd. water is discharged from the bottom. J. S. C. I.

Swedish rosin oil. J. HÜNSCHER. Lab. of the Seifensieder Ztg., Augsburg. *Seifensieder-Ztg.* 48, 231-2(1921).—*Swedish rosin oil, tall oil, pinolein, pine oil, or Swedish olein* is a by-product of cellulose manuf. and has the appearance of a liquid rosin. It may vary according to the kind of wood from which the cellulose is derived. The following results refer to 3 products obtained from one of the factories: (1) *Tall oil*: a dark brown, viscous liquid with an odor resembling rancid lard and contg. a cryst. sediment; sol. in cold alc. but the sediment requires warm alc. for soln; is decolorized by animal charcoal. $D_{15} 0.976$, acid no. 114.5, sapon. no. 129.3, unsaponifiable matter present. (2) *Tall oil distillate*.—This is the product usually offered in commerce; a clear liquid of light yellow color, with an odor similar to the tall oil; it also shows a cryst. sediment. The supernatant liquid has a sp. gr. of 0.958, 1 no. 106.7, acid no. 151.6, sapon. no. 162.2 and 18% unsaponifiable matter. The acid no. of the saponifiable portion is 189.9, the sapon. no. 191.7 and the sp. gr. 0.957, while that of the unsaponifiable portion is 0.974. The unsaponifiable matter is a light yellow viscous oil of resinous odor; the color of the saponifiable portion is orange. The tall oil distillate is readily sapon. with a lukewarm soln. of Na or K carbonate and forms a soft yellow soap of good lathering power and a not unpleasant odor. By grinding 20% of the distillate with 80% soda ash a good washing powder results, but with more than 20% the powder becomes hygroscopic. The Ca soap is a viscous, sticky mass, insol. in alc., but sol. in benzene and petroleum products and would be suitable for plasters, fly paper, etc. The cryst. sediment (4% of the total) is sol. in warm alc. and when twice recrystd. from alc. shows a m. p. of 170° and a sapon. no. of 195.6; it is considered to be pure abietic acid. (3) *Tall oil pitch*.—A glassy, brittle pitch of m. p. 85° (drop-point), sol. in benzene, partly sol. in alc. and ether, burns with slight odor, leaving an ash essentially Fe_2O_3 ; acid no. 50.3, sapon. no. 75.5. It is suitable for insulations and cheap grades of black varnish. Three recipes are appended for utilizing the tall oil distillate as spinning oil, head wash and liquid bath preps. P. ESCHER

Underground conditions in oil-fields. A. W. AMBROSE. *Bur. Mines, Bull.* No. 195, 231 pp. (1921).—A detailed report on the general methods of procedure for studying the underground conditions in oil fields, and a description of the results obtained by proper cooperation between the petroleum engineer and the driller. W. F. FARAGHER

The technical utilization of Swabian slaty marl (Posidonienschiefer) and its commercial significance. G. GRUBE. *Chem. Ind.* 43, 419-24(1920).—This marl is a shale with a content of bitumen of 10-15%. The mineral portion is a clay-limestone, having considerable mechanical strength. Technical operations will be directed to using the lower

portion of the deposits (3-3.5 m. thick), which yields 5-7% of oil on distn. The upper portions of the deposits (4.5 m. thick), yield 3-4% of oil. The reserves of shale in Swabia are estimated at 450,000,000 cu. m., which should yield 50,000,000 t. of crude oil. The heat of combustion of the richer shale is 1000 to 1500 Cal. per kg. Distn. in iron retorts gives only about 40% of the bitumen as crude oil. The low yield is due to the decompn. of the initial oil on the walls of the retorts, since the initial products are very unstable. Vacuum distn. increased the yield of oil considerably (from 7% to 8-9%). Special methods of distn. devised by G. increased the oil from 40% of the weight of the bitumen to 71.3% of this weight. The method (not described) is easily utilisable on a commercial scale. The sulfur content of the shale varies from 1.5-4.7%, present mostly as pyrites. This S can be recovered, giving a yield of 9 kg. per 1000 kg. of shale. The crude oil has a sp. gr. of 0.95, and contains 3-4% of S in org. compds. Distn. gives the following fractions: 5% naphtha; 40% burning oil; remainder in large part suitable for making lubricants. The presence of easily oxidizable unsatd. compds. makes it necessary to exclude air during distn. and refining of the oil. Only small %'s of aromatic hydrocarbons are present. The method of Tausz (C. A. 13, 1148) showed that in the fraction to 165°, the % of unsatd. and S compds. is 65-80%. Production of producer gas from the shale is still an expt. The residue in the retort is used to some extent in making cement and building stone. Gas can be made in regular gas retorts but this process is in the exptl. stage also.

W. F. FARAGHER

Oil production from shale and coal. H. P. BOSTAPH. *Oil Gas J.* 19, No. 50, 64-6 (1921).—General advice is given on the extn. of oil from carbonaceous material such as shales and coal. Using a low temp. distn. process (375-460°) with shales, B. obtained from 20 to 100 gals. oil and from 10 to 60 lbs. $(\text{NH}_4)_2\text{SO}_4$ per ton of shale. Distn. of the oil yields on the av., 8% gasoline, 29.5% illuminating oil, 25.75% gas oil, 6.5% scale, 24.25% tar, 3.25% residue. The gasoline consists of 60-70% olefins, the balance being principally paraffins. The burning or lamp oils are transparent and odorless. The gas oils or intermediate oils have a sp. gr. of from 0.84 to 0.87 and a flash point generally higher than 66°. At a temp. of 800° to 900°, a good gas oil will yield 1200 c. p. per gal. The lubricating oils have a sp. gr. varying from 0.865 to 0.910. These lubricating oils do not decrease as rapidly in viscosity by heating as many mineral lubricants. The paraffin has a m. p. of 43-54°. B. recommends that the usual laboratory fractionation tests be abandoned in favor of a process which would consist in manufacturing the oils into finished products and then examing. them under service conditions.

R. I. SHELLEY

A study of the saturated and unsaturated compounds of shale oils. C. W. BORTIN. *Rail Road Red Book* 38, 803-9(1921); *Chem. Met. Eng.* 24, 876-80.—Shale oils from Colorado were found to contain from 11.5 to 28% saturates as compared with a value of 38% for Scotch shales and 16% for English shales. The av. amt. is about 15.5%. Such a high proportion of unsatn. indicates a lack of H in the org. material found in the shales. Tests carried out on cracking shale oils showed an unexpected increase in satn. in the distd. product and indicated that the high proportion of unsatd. compds. might be due to low-temp. carbonization. Thus the satn. of the crude oil after distn. was as follows: without steam 25.1%, with steam 16.6%, with H 19.6% and at high temp. 30.1%. These oils were then distd. and the oil obtained with steam was cracked to the greatest extent, yielding 10% coke, 2.9% gas, and 86.6% oil containing 35% saturates. The effect of distn. at atm. pressure was studied on an asphaltic oil from a Utah shale, an oil rich in paraffin from Nevada shale and an oil of a mixed base variety from Colorado shale. The vol. of saturates increased (on cracking) 29% for the asphaltic oil, 38.8% for the paraffin base and 73.6% for the mixed base oil. The % of saturates decreases with the increase in gr. and h. p. and reaches a minimum (18 to 40%) in the heaviest fraction.

R. I. SHELLEY

Comparison of Scotch and Colorado shales. R. D. GEORGE. *Rail Road Red Book* 38, 593-7(1921).—A general discussion of the problems of retorting and refining. It is highly probable that modifications of the Scotch retorts and processes may be needed to meet peculiarities of our Colorado and Utah shales but the broad needs are the same.

—Also in *Petroleum Times* 6, 51-2.

R. L. SIBLEY

Studies in Colorado shale oils. ARTHUR J. FRANKS. *Colo. School of Mines. Chem. Met. Eng.* 24, 561-4(1921).—Samples of shale oils were prepd. in an exptl. Ginot retort by low-temp. carbonization of shales found in the vicinity of De Beque, Colo. Distns. were made on 500 cc. samples in a 1 l. flask, the usual 100 cc. flasks proving unsuitable on account of cracking. The distn. fractions were 10% by vol. The action of concd. H_2SO_4 is expressed in terms of "saturation," which is the vol. % of oil unattacked. F. considers the customary statements of results in terms of "unsaturation" as incorrect, inasmuch as many of the products removed by H_2SO_4 , especially in shale oils, are bases, etc., which are not hydrocarbons at all, and are not even unsatd. The samples showed: d. 0.833-0.946, "saturation" 11.7-35.0%, S 0.43-0.64%, initial b. p. 65-90°, coke 5.3-11.8%. The temp. curves are similar in shape, all showing turning points at 350-60°, where cracking becomes marked. Results with two samples representing, resp., the light and heavy worms from the condensers showed that fractional condensation of the retort vapors is not successful, since the light fraction contained a considerable amt. of heavy oils, and vice versa. The lighter distn. fractions were found to be most satd., and satn. decreased with increasing b. p. and d. up to the cracking point, after which the fractions showed both lower b. ps. and higher satn., a phenomenon characteristic of the destructive distn. of Colo. shale oils, but not met with in petroleum distn. It is thought that certain unsatd. resins may yield satd. compds. on cracking.

M. R. SCHMIDT

Oil shale in Colorado. II. R. D. GEORGE. *Rail Road Red Book* 38, 521-5(1921); cf. C. A. 15, 1395.—A series of thin sections from shales giving an oil yield of 90-100 gals. per ton were prepd. and examd. under the microscope. In the leanest shales, the org. materials were very scarce while in the richest shales, the org. matter appeared to be more completely bituminized than in some of the shale having a lower oil content. The best material for microscopic examn. is the dark mahogany brown, faintly lustrous shale. The waxy luster of certain shales is thought not to be due to a paraffin but rather to kaolin or a related hydrous aluminium silicate. The origin of the shales, their structural features and weathering are described.

R. L. SIBLEY

The Freeman multiple retort and Estonian shales. ANON. *Petroleum Times* 5, 432(1921).—A sample of Estonian shale which was soft, light brown in color and readily crushed contained total volatiles 44.5%, fixed C 16.5% and ash 39%. The run on the shale was made in a single chamber retort under agitation and with automatic temp. control and gave as results: total H_2O 7%, residuum 58.94%, 2183 cu. ft. gas and 85.2 gals. oil per ton of av. sp. gr. 0.918. The oil yield is equiv. to 70.14 gals. per ton of dry shale. The oils carry a large % of lubricating base and a high % of gasoline. The oil cracked at 380° at which point 75% of the oil had distd. Of the oil, 20% is a motor fuel averaging 61.9° Bé. The yield of motor gasoline is 14 gal. per ton of dry shale. The gas produced in retorting became highly luminous at about the middle of the oil production. The residue was in the form of hard coke of the compn.: vol. matter 6%, fixed C 50.1%, ash 43.9%. It was an excellent fuel for gas production or for power development.

R. L. SIBLEY

The Freeman multiple retort and Norfolk and Kimmeridge shales. ANON. *Petroleum Times* 5, 416-7(1921).—These results were obtained by low-temp. carbonization in a single-chamber retort under agitation. The temp. of the chamber was regulated by a precision automatic temp. control. The oils obtained in both cases are not emulsions nor do they carry H_2O , tars, waxes, or impurities which render the secondary re-

fining a difficult matter. The Norfolk shale contained 41% total volatile matter, 18% fixed C, and 41% ash. The yields per ton were: total H_2O 13.55%, oil 17.95%, residue 60.71%, gas 7.42% (3500 cu. ft.), loss 0.37%. The sp. gr. of the oil was 0.973, and the yield was 41.3 gals. per ton of raw shale or 47.79 gals. per ton of dry shale. The max. point reached at which distn. of oil ceased was 360° . The Kimmeridge shale contained 52.3% total volatiles, 16.5% fixed C, and 31.2% ash. The yields per ton were: total H_2O 15.47%, oil 20.75%, residue 53.97%, gas 5.8% (2600 cu. ft.), loss 4.01%. The sp. gr. of the oil was 0.973. There was produced 47.76 gals. per ton of raw shale or 56.5 gals. per ton of dry shale. The oil was brownish and apparently high in low boiling fractions. The economical max. temp. is 370° . R. L. SIBLEY

Refractometric determination of paraffin wax as a control test in the manufacturing process. M. FREUND and G. PALIK. *Petroleum* **5**, 757-8(1920).—The proportion of paraffin wax, x , may be calcd. from the refractive index (n_d) of a mixt. of paraffin wax and a filtrate oil, if the respective coeffs. (n_2) and (n_1) are known: $x = (100n_1 - 100n_d) / (n_1 - n_2)$ and conversely, the refractive index of the mixt. may be calcd. when the % of paraffin wax is known: $n_d = [xn_2 - (100 - x)n_1] / 100$. The formulas are only applicable when the sp. grs. of the constituents are practically the same. The results are lower than those obtained by the methods of Holde or Neustadt. J. S. C. I.

Analysis of paraffin wax. K. BUBB. *Petroleum* **5**, 5-8(1920).—The sp. gr. at 50° of lignite paraffin wax increases nearly linearly with the addition of dark paraffin oil, and it has been found that the difference between the sp. gr. at 50° and 0.784 in units of the third decimal place gives the percentage content of dark paraffin oil if hard paraffin wax (m. p. about 50°) is used. With soft paraffin wax and intermediate qualities the figures 0.781 and 0.782, resp., should be substituted. The results are accurate to 1-2% provided that the content of oil is under 44%. The method is useful for controlling the sweating process. J. S. C. I.

Influence of high temperature on paraffin wax. H. BURSTYN and W. JAKUBOWICZ. *Przemysł Chem.* **3**, 36-42(1919); cf. *C. A.* **14**, 3525.—Crude petroleum from Boryslaw, sp. gr. 0.856, after treatment with 50% of 98% H_2SO_4 at 40° and subsequent washing, was distd. According as the distn. was stopped at 150° , 200° , 250° , or 300° the proportion of paraffin wax obtained from the residue was 13.8%, 12.8%, 12.2%, or 10.1%. In each case the paraffin had a setting point of 51.5° . Since no appreciable amt. of paraffin wax passed into the distillates the differences in the amt. of wax obtained are attributed to decomposition, which is stated to occur even at 125° , mainly owing to the action of the air. On this account distn. *in vacuo* is recommended. J. S. C. I.

Determination of paraffin wax in ozocerite. A. S. KOSS. *Przemysł Chem.* **4**, 74-6(1920).—Mix from 2 to 5 gr. of the paraffin distillate with 20% of animal charcoal previously dried at 140° , and ext. the mixt. with petrol of b. p. $50-60^\circ$. Free the ext. containing the total paraffin wax and the paraffin oil from petrol, dissolve in warm acetone, and cool to -22° , whereupon the paraffin wax crysts. while the oil remains in soln. After filtering and washing with acetone at -22° , dry the paraffin at 100° . The soft paraffins sep. from the acetone soln. only at -30° . A sample of ozocerite showed 47.35% of paraffin by the Engel-Höfer method, and 65.85% by the method described above. J. S. C. I.

Testing casinghead gas (THOMPSON) 21. Cold steam distillation (OFFERMANN) 13. Distilling oils, etc. (Brit. pat. 150,774) 13.

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Die natürlichen und künstlichen Asphalte, ihre Gewinnung, Verwendung, Zusammen-

mensetzung und Untersuchung. Edited by J. Marcusson with the coöperation of H. Burchartz and G. Dalén. Leipzig: Wihl. Englemann. M. 25, bound M. 34 + 50%. For review see *Z. deut. Öl-Fett-Ind.* 41, 263(1921).

Petroleum Annual, 1921. Handbook of the Petroleum Industry. Chicago, Ill.: Independent Oil Men's Assoc. 430 pp. For review see *Eng. Mining J.* 111, 756(1921).

23—CELLULOSE AND PAPER

A. D. LITTLE

Cellulose acetate. EDWARD C. WORDEN. *Kunststoffe* 11, 42-45(1921).—Polemical. Cf. *C. A.* 14, 1038.

Chemical engineer and the sulfite division of the newsprint industry. R. W. MCKENZIE. *Can. Chem. Met.* 5, 105-7(1921); *Paper* 28, No. 9, 20-22(1921).—A general discussion of the application of chemical engineering to the paper industry. C. J. WEST

Chemical engineer and the ground wood division of the newsprint industry. R. W. MCKENZIE. *Can. Chem. Met.* 5, 133-4(1921); *Paper* 28, No. 10, 19-21(1921).

C. J. WEST

Digestion of woody fibers with chlorine for the production of cellulose and textile fibers. P. WÄRNIG AND W. GERRISCH. *Text.-Forschung* 2, 69-79(1920).—For the production of cellulose from wood shavings of 0.2-0.3 mm. thickness the authors recommend a preliminary boiling for 1 hr. in 0.1% NaOH soln., then treatment with Cl for several hours without heating, and after the removal of HCl a final treatment in 1% NaOH soln. at the ordinary temp. with stirring. Thick shavings and chips may be digested with Cl under 6 atm. pressure. A preliminary heating with 1% alkali is necessary in the treatment of cereal straws. The Cl process is not suitable for the production of textile fibers from jute and rushes, but it might be applicable to hemp, flax, ramie, and nettles.

J. S. C. I.

Utilization of reeds (*Arundo phragmites* L.). E. HEUSER. *Papierfabr.* 1920, 115. —The cellulose content of the root stems of the common reed (*Arundo phragmites*) was detd. by Cross and Bevan's chlorination method. The cellulose obtained contained 1.09% of ash and 24.74% of pentosan. The chlorination method gives therefore not the percentage of pure cellulose, but of crude cellulose. Calcd. to an air-dried and ash-free basis the following figures were obtained:—25.42% pure cellulose, 44.02% lignin, 14.78% pentosan, 10.68% moisture. By fermentation of the aq. ext. 2 expts. gave an av. yield of 9.28 l. of alcohol from 100 kg. of dry substance. A similar plant, the reed mace (*Typha latifolia*), was worked up in Germany during the war to hemicellulose for cattle food. This hemicellulose had the compn. 53.70% pure cellulose, 28.05% pentosan, 9.45% lignin, and 3.80% ash.

J. S. C. I.

The Rinman modification of the sulfate process. BROR. N. SEGERFELT. *Pulp Paper Mag. Can.* 19, 452-3(1921); *Paper Industry* 3, 96-7(1921).—The R. process is a method of producing cellulose by cooking with weak caustic soln. with the aid of a reducing material, preferably Hg, at a temp. not exceeding 170°. The evapd. black liquor is treated with fresh NaOH and lime, placed in a retort and dry distd. at not over 500°. In order to obtain the highest yield of acetone, MeOH and oils, a small amt. of steam is admitted to the retort. Fuel gases and coke also are obtained and are used to heat the retort. The bad odor of the sulfate process is eliminated, the yield of pulp is higher, and the loss of alkali is reduced to 2-3¼%. Several mills in Germany are using this process and one mill in Scotland is installing it.

H. H. HARRISON

History of the utilization of sulfite waste liquor as a fuel. *Papierfabr.* 19, No. 8, 165-7(1921).—A reprint of an article by Dorenfeld (1897), showing that he had the

same idea that Strehlenert is developing today in his utilization of the organic matter of the sulfite waste liquor as a fuel.

C. J. WAST

Sulfite spirit in 1920. RUDOLF STREHNER. *Papierfabr.* 19, 341-4 (1921).—S. compares the quality of the alc. produced from waste sulfite liquor with that from other sources. There was a marked improvement in the quality of the alc. during the year.

C. J. WAST

Dyeing of straw pulp. HEINRICH PRESS. *Papierfabr.* 19, 261-6, 365-8 (1921).—The straw pulp had the following compn.: cellulose, 40.4%; pentosan, 20.5%; lignin, 29.9%; ash, 9.2%. The ash consisted of 3.45% SiO_2 and 5.15% CaO . The Ca was combined in part as CaSiO_3 and in part with constituents of the straw pulp, perhaps the pentosans. In studying the behavior of dyes, difficulty was experienced in titrating the solns. or in comparing them colorimetrically because of the yellow color of the filtrate from the straw. This was overcome by adding filtrate from the straw (without the addition of dye) to the standard comparison cylinder. The following figures give the % of absorption of various dyes; malachite green extra, 93; rhodamine B, 84; vesuvin Blx, 78; pulverfuchsin AB, 79.5; chrysoidin, 80; naphthol yellow S, 65; pure blue (Reinblau), 75; echtrot, 75; brilliant scarlet, 71; alkali blue, 50; oxydiamine black AT, 95; diamineviolet N, 95. Straw thus reacts towards dyes in the same manner as unbleached pulp. Expts. with colored mineral fillers or pigments showed that the absorption varied from 17 to 57%; this apparently depends upon the state of division of the filler and of the pulp, and is analogous to that of ordinary fillers.

C. J. WAST

Straw lignin. F. PASCHKE. *Wochbl. Papierfabr.* 51, 2322-3 (1920); cf. C. A. 14, 2262.—Treatment of straw with Na_2CO_3 produces good yields of straw pulp if attention is paid to control of temp. and concn. From the alk. ext. a granular reddish brown powder which decomposes at 195° , and has the formula $\text{C}_{17}\text{H}_{10}\text{O}_5$, was isolated. It has a high content of O than wood-lignin. It forms a colloidal soln. with water and is pptd. by solns. of BaCl_2 , CaCl_2 , and other metallic salts. Waste liquors from the treatment of straw with Na_2CO_3 contain no combined NH_3 , as is the case when NaOH is used, which indicates that the protein of the straw is not decompd.

J. S. C. I.

Estimation of mechanical wood pulp. M. G. KOTTEHASKER. *J. Soc. Dyers Colour.* 37, 11-3 (1921).—Methods for detn. of mechanical wood in mixed pulps and papers are classified as: (1) Microscopical, based on color reactions of "chlorozinc-iodine" and aniline sulfate. It is difficult to prep. slides representing av. samples. (2) Colorimetric, based on action of a HCl soln. of phloroglucinol on the pulp, color produced compared with standards. This method is inaccurate for more than 20%. (3) Chemical methods (a) based on fact that mechanical wood pulp will reduce gold chloride while pure forms of cellulose will not (reduced Au is detd. gravimetrically), (b) based on the amt. of phloroglucinol absorbed by the sample. The original and residual solns. are titrated with HCHO and the amt. absorbed is found by difference. The end-point is not sharp. (c) The proposed method based on the substitution of a *p*-nitroaniline soln. (1 g. per l. dil. HCl) for phloroglucinol and after complete absorption the estn. of residual *p*-nitroaniline by filtering through glass wool, and adding to 10 cc. of the filtrate a known excess of titanous chloride, and HCl to prevent hydrolysis, boiling 8-10 min. in an atm. of CO_2 , cooling in tap water, and titrating the residual titanous chloride with standard ferric alum, using KCNS indicator. Air-dry mechanical wood pulp absorbs 0.56-0.57% *p*-nitroaniline. Results correct within 2% error can be obtained. Pure forms of cellulose proved to absorb 0.02-0.04%. The old colorimetric phloroglucinol method is recommended for samples containing less than 20% mechanical wood. The formula for calcg. results obtained by the new method is: % mechanical wood pulp = (% absorption by bone-dry sample - 0.04) \div 0.80.

E. E. FICKERT

Bamboo as a paper-making material. JOHN STRAVENS SRD. *Paper Industry* 3, 84-87

(1921).—Bamboo, pulped by the sulfate process, produces a fiber of the highest quality, the fiber length being only a trifle shorter than sulfite pulp. Using 20% NaOH, 7% Na₂S, cooking for 6 hrs. at a pressure of 90 lb. with 8 lb. of liquor to 1 lb. of bamboo unbleached and bleached yields were obtained of 50% and 44%. The bleached bamboo pulp is particularly adapted for the manu. of book papers requiring a soft, well closed sheet, which is extremely opaque and which bulks well with a high finish.

H. H. HARRISON

Mucilage of parchment paper pulps. F. B. SEIBERT AND J. E. MINOR. *Pulp Paper Mag. Can.* 18, 939-42 (1920). (Cf. SCHWALBE AND BECKER, *C. A.* 14, 837, 3790).—The presence of hydrocellulose is favorable to the development of mucilage during the beating of paper pulp, and this explains why parchment pulps having a high Cu number require less beating than pure cellulose pulps. It is advantageous to subject pulp to a cold acid treatment before beating. This increases the amt. of hydrocellulose present, and also the strength of the paper produced.

J. S. C. I.

Specifications for paper makers' quicklime and hydrated lime. Bur. Standards, *Circ.* 96(1920).—The specifications given have been approved by the National Lime Assoc. and the Technical Assoc. of the Pulp and Paper Industry (U. S. A.). The percentage comps. specified are: Quicklime; CaO, 95 (min.); MgO, 2 (max.); insol. matter including SiO₂, Fe₂O₃, and Al₂O₃, 2 (max.); CO₂, 2 (max.). Hydrated lime: CaO, 72 (min.); MgO, 1.6 (max.); insol. matter, including SiO₂, Fe₂O₃, and Al₂O₃, 1.6 (max.); CO₂, 1.6 (max.). For analytical methods cf. Hillebrand *C. A.* 14, 389.

J. S. C. I.

The new Hall process of grinding pulp. W. A. MUNRO. *Pulp Paper Mag. Can.* 19, 446-7 (1921); *Paper Industry* 3, 119-21 (1921).—The dressing put on the surface of the stone is the important feature of this process, which produces ground wood pulp of such high quality that 50% of the sulfite used in newsprint can be omitted. Papers made of pulp from this process have been successfully used in newspaper press rooms.

H. H. HARRISON

Bleaching powder and electrolytic bleach for paper half-stuff. G. SCHUMANN. *Papierfabr.* 19, 287 (1921).—The use of the one or the other of these bleaching agents will depend upon the cost of the power required for their production.

C. J. WEST

New tearing tester. R. MARX. *Paper Makers' Monthly J.* 59, 151-4 (1921).—Discussion of the Elmendorf paper tester.

C. J. WEST

Chemical standards for the technical valuation of raw materials. McDONALD. *World's Paper Trade Rev.* 75, 1042, 1172 (1921).—Discusses, in particular, methods of testing wood pulps.

C. J. WEST

Control of alum content of the tub-sizing bath. P. W. CODWISER. *Pulp Paper Mag. Can.* 19, 499-501 (1921).—A sample of size is measured into a small beaker, heated to 130° F. on the water bath and tested by immersing a small strip of yellow bromothymol blue paper in it. If the color of the paper is unaffected the titration with 0.1N NaOH is commenced and continued with vigorous stirring until a fresh strip of the paper is turned to a distinct green; it is finished by continuing until a blue-green reaction is obtained. Since the end-point is considerably beyond that at which a green becomes noticeable, some difficulty with low results may be experienced at first. The color changes produced in the paper will alter on standing and partial drying. It is possible to use litmus but results are decidedly below the correct ones. A correction factor must be detd. to take care of the error inherent to an indicator having such a working range when used with Al₂(SO₄)₃. Sample analyses show that this method is good to approx. 8% of the alum present.

H. H. HARRISON

Resin or engine sizing. C. C. SCHNEIDER. *Paper Trade J.* 72, No. 18, 48-50 (1921).—Expts. by S. show that proper emulsification of size is as important as high content of free resin. With hard water the emulsion after standing a little while, will deteriorate

and for good sizing the temp. must not rise above 100° F. At this temp. the aluminium resinate starts to solidify, losing its fine flocculent form, gathering into bigger lumps and forming metallic rosin.

H. H. HARRISON

The rational theory of the beater. SIGURD SMITH. *Papierfabr.* 19, 1-7, 300-316 (1921).—A mathematical treatment.

C. J. WEST

Work on paper testing published by the Materialprüfungsamt. W. HERZBERG. *Papierfabr.* 19, 317(1921).—Subject index of the work from 1914 to 1919. C. J. WEST

Action of hydrogen peroxide on cellulose (HALLER) 25. Determination of the total sulfurous acid in organic substances (FROBESER) 7.

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Cellulose compounds. DEUTSCHE CELLULOID-FABRIK. Ger. 332,203, Jan. 10, 1918. Mercerized cellulose is made to enter into reaction, in presence of caustic alkalies, with $\text{CH}_3\text{ClC}_2\text{H}$ or its homologs. Cf. C. A. 14, 1755.

Removal of noxious substances from waste gases of cellulose manufacture. METALLWERKE VORM. J. ADERS, A.-G. Norw. 30,632, Apr. 12, 1920. Into the gas current small bits of wood, sawdust or other ligneous waste are introduced.

Freeing sulfite liquor from salts. ZELLSTOFF-FABRIK WALDHOF UND OTTO LCHRS. Norw. 30,733, May 10, 1920. The sulfite waste liquor is freed of lime by agents which practically entirely remove the CaO (hence not as sulfate), then concd. and mixed with H_2SO_4 to convert the remaining bases into sulfates, which will sep. out as such upon cooling.

Purifying alkaline waste liquors from disintegration of cellulose polluted and colored by organic admixtures. FA. FR. KÜTTNER, ERNST PROFELD and EMIL SIDLER. Ger. 322,461, July 8, 1919. The waste liquors are inspissated in an open vessel and simultaneously oxidized until the org. substances sep. out. The liquor is preferably heated, while air is being blown in, until the org. substance carbonizes. Oxidation agents, such as saltpeter, may be added until the org. substance seps. out in flaky form and, upon heating for some length of time, in the form of C. Or the liquor may be concd. with solid NaOH to about 30° Bé., then oxidized and heated until the org. material or C seps. The liquor is then decanted or filtered. The process is stated to be particularly applicable to waste liquors from mercerizing processes and viscose manuf.

Working up waste liquors from soda cellulose manufacture. HEINRICH ACHENBACH. Ger. 322, 771, Feb. 16, 1919. The waste liquors are subjected to preliminary thickening to about 10° Bé. by means of fire gases in a cooling tower. The black liquors are as a rule greatly polluted by washing lyes, so that it is not concd. waste liquors from the digesters but mixed liquors of boiling lyes and washing lyes down to 5°

Bé. that are evapd. The waste liquors are boiled down in a cooling tower by fire gases under continuous circulation from about 5-7° Bé. to 9-10° Bé. and then kept at that degree of concn. by regulating the admission of fresh liquor and the discharge of thickened liquor. While being boiled down to this density, the liquor has given off most of the volatile substances and has thus lost its tendency to froth, while it continues to show good trickling power.

Process for rendering sulfite waste liquor suitable for further treatment. ERIC ÖMAN. Addition to Norw. 28,888, June 7, 1920. The process described in Norw. 28,888 for salting out salts of lignin acids from sulfite waste liquor is modified by sepg. into salts of ligninsulfonic acid which are pptd. and filtered off, and those which remain in soln. in the filtrate.

Treatment of waste sulfite liquors. RALPH HARPER MCKEE. Norw. 30,706, May 3, 1920. To free sulfite-cellulose waste liquors of sulfites by means of Ba compd., prior to fermentation to alc., the Ba compds. are used in combination with simultaneous oxidation, in order that the sulfites may be sepd. out as BaSO₄. Cf. C. A. 15, 1076.

Increasing the softness and suppleness of shaped articles produced from cellulose ethers. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. Ger. 322,619, Mar. 29, 1917. —Ethyl resorcinoldicarboxylate, which is insol. in H₂O and boils at 298-302°, is adapted to increase the softness and suppleness of shaped articles produced from cellulose ethers. Example: 10 parts by wt. of ethylcellulose are dissolved in 100 pts. by wt. of acetone, and to the soln. 5 pts. by wt. of Et resorcinoldicarboxylate are added. The soln. is then poured upon a horizontal glass plate. After evapn. of the solvent there remains a white flexible plate. Other cellulose ethers such as benzylcellulose or the like, may also be used, and for the acetone, acetic ether may be substituted.

Production of parchment-like paper. PAUL HÖRRING. Ger. 303,266, Sept. 1, 1916. Leaves or stalks, or both, of reed mace are subjected to the action of alkali lye under pressure. The resultant product, after having been washed, is used as whole-stuff for paper manuf., without special grinding and without sizing. A very translucent, fat-proof and ink-resisting sheet of parchment-like character of brownish yellow color is produced. Before being made into sheets, the paper pulp may be bleached with chloride of lime without impairing its strength.

Treatment of tang. A. S. TANGPAPIR. Norw. 31,416, Nov. 22, 1920. In order to render tang suitable to be worked up into paper pulp, it is treated in fresh moist state after having been comminuted, with 4 kg. of Ca(OCH₃), 1 kg. of CaO and 1/2 liter of H₂SO₄ to each 50 kg. of tang. It is then treated with (HOCO)₂ and after being removed from the liquor and rinsed out, it is put in the hollander after addition of Na₂CO₃.

24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE

The detonation of explosives. H. KAST. *Z. ges. Schiess-Sprengstoffw.* 15, 195-7 (1920).—The effect of the air enclosed in a compressed charge of explosive was considered in connection with the various sensitiveness of explosives to propagation of detonation. TNT was compressed to different ds. and the air content of the pressed charge detd. by melting in hot, air-free H₂O and collecting the liberated air in a measuring app. The difference in air content of charges varying from 1.47 to 1.59 in d. was slight; a 50 g. charge at d. 1.59 contained 0.7 cc. air, and one at d. 1.47 only 2 cc. Rate of detonation of TNT at ds. varying from 1.31 to 1.59 was detd. in evacuated iron pipes at pressures of 5-7 mm. Results were normal, agreeing with those at atm. pressure. Detonation is thought to be the result of mol. rearrangement brought about by dynamic pressure. The effect of phys. structure on ease of detonation is apparent in the result

of hardening of plastic gelatin dynamites and cheddites on storage, and in the case of explosives alike chemically but of different structure; *e. g.*, guncotton with 16% H_2O detonates at 6800 m. per sec., dry guncotton at 6300 m., while nitrocellulose smokeless powder, chemically practically the same as guncotton, cannot be detonated in compact condition, and in granular form transmits detonation at only 1000-1800 m. per sec.—rather a rapid burning than detonation. Tables of results are given showing rates of detonation of nitroglycerin, blasting gelatin and dynamites. The rate for black powder in iron pipe 32-41 mm. diam. and 6.3 mm. thick was 420 m. with fuse and 382 m. with 2 g. detonator.

C. G. STORM

Mercury fulminate and its solvents. A. LANOHANS. *Z. ges. Schiess-Sprengstoffw.* 15, 219-21, 227-9, 235-7 (1920); cf. *C. A.* 14, 3531.—Inorg. solvents for $HgC_2N_2O_4$ (A) are KCN, NH_4OH , $Na_2S_2O_3$, and KI; org. solvents are pyridine and methylamines. (1) KCN solns. of A give a ppt. with HNO_3 which is pure A and not an addition compd. (cf. *C. A.* 4, 1101, and *Ber.* 9, 779). Gray fulminate gives a pure white ppt., showing that $HgCl$ is not accountable for the white color since this impurity would not be pptd. by HNO_3 . Contrary to Solonina (*C. A.* 4, 1101), L. found that KCN solns. of A were not stable, turning brownish to red-violet and forming a surface scum. Heating hastens the decompn. After 10 to 14 days, resp., heated and unheated solns. could no longer be pptd. by means of HNO_3 or H_2SO_4 . Cu, Zn, Al and brass were rapidly attacked by KCN solns. of A; Ni, Fe, and Sn were not affected. NH_4SCN forms a cryst. double salt with A in KCN soln. NH_4NO_3 and $(NH_4)_2SO_4$ are without effect. (2) NH_4OH : By dilg. a satd. soln. of A in 22% NH_4OH , large pure white crystals were obtained, even from gray fulminate. On treating with H_2O , these crystals become opaque, indicating decompn. and leaving doubt as to whether the ppt. was pure A or an additional compd. with NH_4 (*Ber.* 7, 1244; 8, 518, 1177). Addition of HNO_3 , H_2SO_4 or $CuSO_4$ also causes pptn. of A from soln. in NH_4OH . At atm. temp. about twice the concn. of NH_4 is necessary for soln. as at 30°. The soln. of A in NH_4OH decomposes completely in 12 hrs. standing, forming ppts. of various colors (red, yellow, white and blue), contg. only traces of A. No definite compd. of Hg was isolated from the ppt., which contained 80-86 % Hg. (3) *Amines*: $MeNH_2$, ME_2NH , and ME_3N in 33% solns. dissolve A less readily than NH_4OH , and with sepu. of more free Hg. On diln., pure white A was pptd. On long standing, these solns. decompose, forming sediments contg. metallic Hg. After 10 days, filtrates from all of the solns. gave negative tests for Hg. (4) $Na_2S_2O_3$ is not a solvent for A but forms a double salt. The alkalinity of the soln. also indicates decompn. A 10% soln. of $Na_2S_2O_3$ attacks A most readily, 600 cc. readily dissolving 30 g. in a few min. (5) *Aniline*: Contrary to the results of Steiner, who found that $PhNH_2$ caused violent decompn. of A, (*Ber.* 7, 1244), L., by the use of a large excess of $PhNH_2$ (20 : 1), found no energetic action. The A dissolved, leaving a gray deposit of Hg; the filtered soln. changed from yellow to dark red in 3 hrs., then began to deposit Hg, becoming opaque in 24 hrs. (6) *Pyridine*, unlike $PhNH_2$, is a true solvent for A. Pure A is pptd. on diln., even after 24 hrs. If A is prepd. from EtOH denatured with pyridine the yield is poor but the product is more explosive. A is not pptd. from pyridine solns. by HCl, KI or I solns. Long heating on a steam bath or long standing in a closed vessel at room temp. causes decompn. with sepu. of a ppt. contg. about 58% Hg. Other acids than HCl ppt. pure A. (7) *Other solvents*: A is slightly sol. in EtOH, Me_2CO , insol. in C_2H_6 , $CHCl_3$, glycerol, epichlorhydrin, linseed oil, and turpentine. (8) L. found that if A is dissolved in 18% HCl (3 g. in 50 cc.), and the soln. titrated with normal KI soln., the result indicated that only about 87.5% of the Hg existed as Hg_2Cl_2 , the remainder as $HgCl$, and suggests this as a method for detg. the Hg content of A, correcting for the 12.5% of Hg, which does not react with KI (theoretically 1 cc. normal KI = 0.071 g. A). A number of photomicrographs show crystals of A from the various solvents.

C. G. STORM

A new explosive, umbrite; its use in agriculture. G. TOMMASI. *Ann. stas. chim. agrar. sper.* II, 9, 75-84 (1917-1919).—Umbrite A, contg. 49 pts. of nitroguanidine, 38 of NH_4NO_3 , and 13 of Si is suitable for artillery shells, while umbrite B, contg. 37.5 pts. of nitroguanidine, 49.4 of NH_4NO_3 , and 13.1 of Si, which has a greater disruptive power, serves well for bombs, for mining purposes and for loosening the soil. These explosives are but slightly hygroscopic and retain unaltered their explosive power even when they contain a certain proportion of water. J. S. C. I.

Detection of nitrous compounds and determination of aromatic nitro-derivatives in smokeless powders. CORRANO LUTRI. *Giorn. chim. ind. applicata* 2, 557-9 (1920).—L. found that a good test for nitrous compds. in smokeless powders is furnished by a 1% AcOH soln. of α -naphthalamine, which gives a characteristic intense violet color, not only with HNO_2 but with nitrosamines and nitroso derivs. His method of detg aromatic nitro-derivs. in smokeless powder is as follows: Reduce 3 g. explosive to powder (preferably by rasping) ext. for about 6 hrs. with Et_2O , evap. the solvent at low temp. and treat the residue in a flask provided with reflux condenser, with 50 cc. 20% H_2SO_4 , warming on the bath and agitating as much as possible for about 6 hrs. In the cooled acid liquid may be noticed drops of the solidified nitro-compd. Sep. these by filtration. Ext. the filtrate with C_6H_6 and evap. the solvent by warming at about 50° in a tared dish. Induce crystn. of the residue with a trace of solid dinitrotoluene, add the substance already sepd. by filtration and weigh. L. made the detn. of dinitrotoluene in several ballistics, and of trinitrotoluene in other explosives, with satisfactory results.

ROBERT S. POSMONTIER

Determination of the water content of moist nitrocellulose. A. LOGOTHETIS AND G. GARGOROUPOULOS. *Z. ges. Schiess-Sprengstoffw.* 16, 1-2 (1921).—A simple, rapid, safe, and efficient control method for detn. of H_2O in wet nitrocellulose consists in mixing the wet sample with alc. and from the extent of diln. of the latter, calcg. the H_2O content of the sample. A weighed sample of the wet nitrocellulose (1 part) is mixed with a measured vol. (about 5 parts) of EtOH , approx. 96% by vol. and of known sp. gr., the mixt. well stirred, filtered, and the sp. gr. of the filtrate detd. The wt. of H_2O taken up by the known vol. of EtOH is then calcd., allowance being made for contraction in vol. on diln.; this is then the wt. of H_2O in the sample of nitrocellulose. The method is applicable to all grades of nitrocellulose and checks closely with the usual method of oven-drying to const. wt. C. C. STORM

Report on explosions which occurred during the war in the Sieburg Fireworks Laboratory. A. LANGHANS. *Z. ges. Schiess-Sprengstoffw.* 16, 2-3, 10-2, 17-9, 35-7 (1921).—A detailed account of numerous large and small explosions which occurred in the manuf., and loading of mercury fulminate, primer compns., primers, light compns., fuses, tracers, etc., contg. many items of interest to manufacturers of these materials. C. C. STORM

Determination of nitrous fumes in air, with special reference to fuse igniters (MOIR) 7. The presence of nitrates in the leaves of trees growing near picric acid works (BURRELL, DOUGLAS) 11D. Drying colloids (Brit. pat. 159,465) 13. Technical production of picric acid from benzene (ПРОСВѢЖКА) (БЛЕЧТА) 10.

СООРНА-КЛУ, А.: The Storage of Petroleum Spirit and Carbide of Calcium. Revised Ed. London: Charles Griffin & Co. For review see *Chem. Trade J.* 68, 634 (1921).

25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Simple tests for proving the value of aniline oil. RAFFAELLE SANSONE. *Am. Dyestuff Reporter* 8, Sec. 2, 26-8(1921).—The tests should be regulated in accordance with the purpose for which the oil is to be used. In all cases these tests will be much simplified if a works standard is fixed for the oil or aniline salt, and all new deliveries are tested against this standard, a few carefully chosen and accurately executed tests yielding the required information. A fixed standard established by tests on a factory scale should not be changed until, by thorough practical trial in the works, a change is proved advisable. The tests and methods recommended are (1)—*Sp. gr.* by the hydrostatic balance or the bottle. (2) *Boiling point and percent of pure aniline oil.*—Distill 100 cc. until the temp. reads 182°, change the receiver and continue the distn. until the temp. reads 184°, the fraction collected between these temps. will be pure aniline. (3) *Coloration test.*—To 2 portions, each containing 1000 cc. of water and a fixed amt. of fresh NaClO, is added exactly 1 g. of the works standard and 1 g. of the oil to be tested, resp. After thorough agitation and allowing to stand the colors are compared. (4) *Non-basic substances.*—Treat 10 cc. of the oil with 50 cc. water and 40 cc. HCl, any turbidity indicates the presence of hydrocarbons or of nitrobenzene. (5) *Moisture.*—The first 10 cc. which pass over on distn. are placed in a 15 cc. graduated cylinder and agitated with 1 cc. satd. salt soln., the diminution of vol. indicated the quantity of water present. (6) *Sulfur.*—One hundred cc. of the oil are boiled under a reflux condenser, a stream of CO₂ is passed through the boiled aniline and then through N AgNO₃ soln., a black ppt. indicates S in the oil. Details are given for performing trials on a factory scale.

L. W. RIGGS

Obtaining azo dyestuffs by coupling diazo compounds with certain oxazine and chrome mordant dyes. E. JUSTIN-MUELLER. *Rev. gén. mat. color.* 24, 130(1920).—Diazotized *o*-naphthylamine, ethoxybenzidine, or *p*-nitraniline may be coupled with such oxazine dyes as Gallocyanine, Modern Violet, and phenocyanine and with azo chrome mordant dyes (diazotized benzidine sulfonatedisulfonate may also be used) to produce dyestuffs sparingly sol. in water but sol. in NaHSO₄, and suitable for printing. A suitable printing paste is made by allowing 3 pts. of dyestuff with 2 pts. of NaHSO₄ of 38° B. (sp. gr. 1.36) to stand overnight, adding 2 pts. of glycerol in 24 pts. of gum soln., warming, cooling and adding 6 pts. of Cr acetate of 20° B. (sp. gr. 1.16). The printed cloth is steamed for 1 hr. soaped at 80° and dried. The moss green to marine blue effects so obtained are fast to washing, soaping, air, and light.

J. S. C. I.

Production of lakes of basic colors with zinc ferrocyanide. E. JUSTIN-MUELLER. *Rev. gén. mat. color.* 24, 129-30(1920).—Color lakes suitable for printing on paper and textiles are obtained by mixing solns. of a basic dye (1 pt.) and ZnSO₄ (2 pts.), and then adding a soln. of Na or K ferrocyanide (2 pts.).

J. S. C. I.

Method for printing color reserves under sulfur colors with vat dyes. L. DISHERENS. *Rev. gén. mat. color.* 24, 113-5(1920).—Cloth is printed with a paste contg. per 1000 pts., 150 pts. of Ciba Blue 2B 20% (or other vat dye), 250 pts. of hydrosulfite-sulfoxylate 36%, 250 pts. of British gum, 250 pts. of ZnCl₂, and 100 pts. of NaOH of 38° B. (sp. gr. 1.36), dried, twice steamed in a Mather-Platt for 4 mins., dyed in a bath contg. per 1000 pts., 40 pts. of Immedial Carbon BN, 40 pts. of Na₂S, 10 pts. of NaOH of 38° B. (sp. gr. 1.36), and 40 pts. of castor oil soap, then immediately washed, soured, washed, and soaped. Heavy ground shades may thus be obtained. It is important that the printing paste contain the dyestuff in the unreduced form, and in suitable cases Zn sulfoxylate may be used in place of the two sep. components; the alkali employed may then be NaOH, borax, or AcONa. S dyes may be used in the reserve paste if

they be only slightly sensitive to $ZnCl_2$. Less satisfactory results are obtained by the use of Fe sulfate instead of $ZnCl_2$, but the dyestuff in its reduced form may then be present in the printing paste.

J. S. C. I.

Printing fabrics with basic dyestuffs. A. STIEGLER. Sealed note No. 2085. *Bull. soc. ind. Mulhouse* 86, 173-4(1920); Report by C. SUNDER. *Ibid* 174-175.—The fixation of basic colors in printing may be advantageously carried out by using Pb acetate instead of Sb salts. Cloth is printed with a paste contg. 30 g. of basic dye, 100 g. of lactic acid, 500 g. of starch-tragacanth thickening, 180 g. of 50% tannic acid soln., 20 g. of Pb acetate, and 170 g. of water; steamed for 1 hr., washed and soaped, and finally chemicked. The lactic acid prevents the formation of the Pb color lake in the printing paste. The colors so obtained are pure, bright, and as fast to washing and light as those obtained with Sb salts. The shades are also heavier. For naphthol-prepd. cloth, a steaming of 5 min. is sufficient. Sunder reports favorably on the process but owing to the sensitiveness of Pb salts to H_2S , suggests that Sb salts be used in the soap bath.

J. S. C. I.

Process for printing reserves under Indanthrene Blue. J. POKORNY. Sealed notes 2180-1 and 2186-7, 23.5 and 26.6.12. *Bull. soc. ind. Mulhouse* 86, 257-8(1920); Report by P. BINDER, *Ibid* 258-9.—Cotton fabric is printed with a reserve paste, contg. $MnCl_2$ without the addition of a dichromate, dried and dyed with Indanthrene Blue. A suitable paste for white reserve consists of 370 pts. of $MnCl_2$, 550 pts. of gum tragacanth, and 80 pts. of water. For red reserves on cloth prepd. with β -naphthol the paste contains 293 pts. of $MnCl_2$, 509 pts. of gum tragacanth, 165 pts. of a soln. of diazotized *p*-nitro-*o*-anisidine, and 33 pts. of $AcONa$. A paste suitable for yellow reserves consists of 240 pts. of $MnCl_2$, 200 pts. of Indanthrene Yellow R. paste, 500 pts. of gum tragacanth, and 60 pts. of water. The addition of 150 pts. of $SnCl_2$ to 1000 pts. of one of the above pastes, allows reserves under Indanthrene Blue and overprinted azo dyes to be obtained simultaneously. Binder reports that the process works well.

J. S. C. I.

Abridged method of dyeing with aniline black. HENRI SCHMID. Sealed note No. 473, Aug. 12, 1886. *Bull. soc. ind. Mulhouse* 86, 397-400(1920).—For dyeing piece goods the machine should be constructed so as to require but a small vol. (perhaps 3 l.) of the dye soln. in which to work. In prep. the bath 2 solns. are made up as follows: (A) aniline-HCl cryst. 2.5 kg., aniline oil 20 g., warm water 5 l.; these are mixed, 5 l. of warm water containing 1 kg. $KClO_3$ are added and the whole is made up to 20 l. with cold water. (B) $Fe(NO_3)_3$, 48° Bé., 1 l., HCl 21° Bé., 1.5 l. and water to make up 5 l. Eight l. of A are mixed with 2 l. of B. The piece is passed once through the bath and allowed to remain rolled up 18 to 36 hrs. when a greenish black is formed, which is finished with chrome or by ordinary means. For producing aniline bronzes 30 kg. of aniline-HCl cryst. are dissolved in 40 l. of water, 1 kg. aniline oil is added, then 8 kg. $KClO_3$ and 4 kg. $NaClO_3$ dissolved in water and the whole made up to 200 l. After cooling, to 80 l. of this mixt. are added a mixt. consisting of HCl 21° Bé., 1.5 l., $Fe(NO_3)_3$ 40°, 200 cc. and water 3 l. The pieces are passed on the jigger and allowed to roll up without squeezing. The goods are unrolled and rolled again without passing through the bath, then allowed to stand 18 to 36 hrs. having care to keep them well covered. When oxidation is completed and the goods are rinsed with water, free of Ca, a steel blue is produced. Treated with Na_2CO_3 the blue passes to the reddish brown of Cu; chromate gives an olive. Report on sealed note no. 473 of Henri Schmid. TH. STRICKER. *Ibid* 400-1.—Stricker questions the utility of Schmid's process on account of the heat produced during the oxidation in the wet rolls, which might tender the fiber.

L. W. R.

Dyeing with direct and substantive colors. RAFFAELE SANSONE. *Textile Am.* 33, No. 6, 28-9; 34, No. 2, 17-8; No. 3, 21(1920).—Directions are given for handling cotton

fabrics continuously through the operations of padding, stentering, washing, souring, rinsing, neutralizing, dyeing, rinsing, drying and finishing, all through one series of machines in the same room.

CHAS. E. MULLIN

Production on the fiber of a deep blue resembling vat blue. HENRI SCHMID. Sealed note No. 997, Sept. 11, 1897, addition to Sealed note No. 922, Oct. 28, 1896. *Bull. soc. ind. Mulhouse* 86, 401-2(1920).—By the revised formula the bath should contain $K_4Fe(CN)_6$ 14.5° Bé., 50 l., *o*-toluidine-HCl 6.5° Bé., 12 l., Na_2SO_4 7.5° Bé., 22 l., $NaClO_2$ 17° Bé., 8 l., HCl 14.5° Bé., 8 l. Report on sealed notes nos. 922 and 997 of Henri Schmid. TH. STRICKER. *Ibid* 402-3.—The process depends on the oxidation of the *o*-toluidine by chlorate and ferrocyanide. An examn. of the samples accompanying the notes and repetition of the expts. showed that the color was largely Prussian blue. By the modified formula the color was not a deep blue properly speaking but a bluish black or violet containing a certain proportion of Prussian blue and becoming gray by alkaline treatment.

L. W. RIGGS

Absorption of coloring matters by charcoal. E. KNECHT. *J. Soc. Dyers and Colourists* 36, 201(1920).—A reply to the paper of Philip, Dunnill and Workman (*C. A.* 15, 1395), in which the paper of Knecht and Hibbert (*C. A.* 11, 2852) is criticized.

L. W. RIGGS

Method for printing resists under formaldehyde-sulfoxylate discharges. E. JUSTIN-MUELLER. *Riv. gen. mat. color.* 24, 129(1920).—Provided that no caustic soda be present, Na, K, or NH_4 ferricyanide is a satisfactory resist. Dyed cloth is printed with a paste containing 200-300 g. of $K_3Fe(CN)_6$, 600 g. of water, and 400 g. of sol. starch, dried, printed with a discharge paste contg. hyraklit C. W. extra, dried, steamed in the Mather-Platt, and washed.

J. S. C. I.

Discharges with formaldehyde-hyposulfite on united dyes in colors directly diazotizable by printing of amines resistant to the reductor. ANDRÉ HUG. Sealed note No. 1596, Jan. 23, 1906 (part 1). *Bull. soc. ind. Mulhouse* 86, 408(1920).—Example: Dye with diamino blue of Cassella; print with a dye containing primuline and aldehyde-hyposulfite; steam 4 min., pass through a bath of H_2SO_4 , add $NaNO_2$, rinse and couple with β -naphthol.—Report on sealed note No. 1596 (part 1) of André Hug. PIERRE BINDER. *Ibid* 408.—For priority cf. *Färber-Ztg.* 1900, 48, and *Treatise on the benzidine colors*, Fr. Bayer and Co., edition of 1898, page 45.—Hug's procedure differs only in the use of aldehyde-hyposulfite in place of Zn powder and bisulfite.

L. W. R.

Production of white and colored discharges on tannin-antimony mordanted cotton. J. POKORNY. Sealed note 2063, 25.1.11. *Bull. soc. ind. Mulhouse* 86, 260-1(1920). Report by P. BINOER, *Ibid* 261-2.—Cotton fabric mordanted with Sb tannate is printed with a thickened soln. of a vat dyestuff, overprinted with a discharge paste contg. NaOH, steamed in a Mather-Platt, washed, soaped, and dyed with basic dyes. The NaOH simultaneously fixes the vat dye and destroys the mordant. Improved effects are obtained by incorporating glucose with the mordant, by chroming after steaming, and by adding about 50 g. of an alc. soln. of β -naphthol to the vat dyestuff. Binder reports favorably on the process, which has been previously suggested for discharges on indigo. The temp. should not exceed 30-50° while after-chroming, in order to avoid dulling the colored ground.

J. S. C. I.

Methods for discharging indigo blue with glucose. A. SCHREUNERT AND N. WOSNIEWSKY. Sealed notes 1990, 6.4.10; 1994, 22.4.10; 2019, 20.7.10; and 2075, 15.3.11. *Bull. soc. ind. Mulhouse* 86, 263-6(1920). Report by M. BATTEGAY, *Ibid* 266-7.—White discharge effects are obtained by printing a fabric dyed with indigo blue, with a paste contg. 300 g. of glucose, 700 g. of thickening, and (optional) 100 g. of ZnO and 50 g. of Leucotrope W, passing it through a soln. of NaOH of 30° B. (sp. gr. 1.26) at 100-110°, and immediately through boiling water. A short steaming of the fabric pre-

vious to treatment with NaOH improves the result. For colored effects the discharge paste contains 250 pts. of a suitable dye, 300 pts. of glucose and 450 pts. of thickening. The process may be combined with the production of ingrain dyes, the discharge paste then contg. 200 g. of glucose, 150 g. of a soln. of diazotized α -naphthylamine, chloroanisidine, or benzidine, 50 g. of Leucotrope W, and 600 g. of thickening, and being used on indigo-dyed cloth prepd. with β -naphthol. A brilliant red discharge is obtained by use of a paste contg. 250 g. of a soln. of diazotized Azo Rose BBI, 200 g. of glucose, 500 g. of British gum, and 50 g. of Leucotrope W. The printed material is passed through a NaOH bath at 100°. If 50 g. of SnCl_2 be added to the above discharge pastes, the quantity of glucose used may be reduced to one-half, the NaOH soln. may be at 20–25° B. (sp. gr. 1.16–1.21) and the resulting colors are more brilliant and intense. Battagay reports that the processes are better than the usual one in which the discharge paste consists of a mixt. of glucose and NaOH

J. S. C. I.

Discharges on aniline black of amines not reducible by diazotizing. ANDRÉ HUG. Sealed note No. 1596 (part 2) Jan. 23, 1906. *Bull. soc. ind. Mulhouse* 86, 409(1920).—Example: Primuline is printed on non-developed Prud'homme black with the addition of aldehyde-hyposulfite. After steaming in a small Mather-Platt the goods are passed into a soln. of NaNO_2 and H_2SO_4 containing dichromate, or if preferred, BaO_2 or Na_2O_2 , washed, coupled and soaped. Result: primuline red discharged upon a black foundation. The printing color should contain primuline 100, glycerol 60, tragacanth satd. in NaOH 600, sodium phosphate 100; these are dissolved with heat and added to warm Rongalite C(BASF) 50 to 80, benzene 20 and the whole made up to 1000. The developer should contain NaNO_2 5 g., H_2SO_4 52Bé. 10, water 985. Report on sealed note No. 1596 (part 2) of André Hug. PIERRE BINDER. *Ibid* 410.—The priority and utility of Hug's process are allowed. Binder recommends that the printing formula be revised to read, primuline 100, glycerol 60, tragacanth 60% 500, NaOH 38 Bé. 50, sodium phosphate 100, Rongalite C 100, benzene 20; and the developer BaO_2 5 g., or Na_2O_2 5, or $\text{Na}_2\text{Cr}_2\text{O}_7$ 10 per liter.

L. W. RIGGS

Dyeing of hair and felt. ALBERT WINTER. *Deutsche Färber-Ztg.* 57, 316–7(1921).—

In the dyeing of loose hair material the salts such as Na_2SO_4 , NaCl, tartar preps., etc., should not be added to the bath as they impair the felting properties of the fiber. Instead of the salts each liter of the bath should contain AcOH 1–2 g., NH_4AcO , 10 Bé., 5 to 10, or H_2SO_4 , 66%, 1 g. After dyeing the goods are felled in dil. H_2SO_4 or boiling dil. AcOH. Colors must be selected that will withstand the acid in the fulling process and be fast to alkali, ironing, steaming and light. Generally substantive and mordant dyes are used, the former being especially adapted to dyeing prepd. wools. With chrome mordant the bath should contain 3 to 6% K_2CrO_4 and 3 to 6% $\text{KHC}_4\text{H}_4\text{O}_6$. Goods are entered at 35°, warmed slowly to boiling and boiled until the bath is exhausted.

L. W. RIGGS

Fastness requirements of dyestuffs by the silk industry. WALTER M. SCOTT. *Am. Dyestuff Reporter* 8, Sec. 2, 11–14(1921); cf. *C. A.* 15, 1219.—The tests which the dyestuffs should stand depend upon the usage for which the finished products are intended. From this standpoint silk goods may be classified into (1) dress goods, (2) upholstery fabrics, (3) decorative goods, (4) ribbons including neckties, and (5) hosiery. Fastness to light is graded as *excellent*, which is equiv. to no change when exposed to 100 hrs. of June sunlight; *fair*, when not more than 50% of the color is lost by such test, and *poor*, when more than 50% of the color is lost. Fastness to washing may be standardized by using a 1% soap soln. in which dyed samples are immersed for 5 min. at 60 to 100°. Those dyes which show no appreciable loss being classed *excellent*, those which show no loss when tested as above but at 33° to 49° are called *fair*, and dyes which do not pass the latter test, *poor*. Changes of cast in artificial light must receive attention in selecting dyes for fabrics which form any part of a color scheme.

Generally it is best to use only such dyes as maintain the same shades under natural and artificial lights and especially to avoid any which tend to redden under the latter. Tendency to crock or rub off is tested by dyeing a sample a high percentage (5 to 10%) and rubbing a moist piece of white silk or cotton vigorously across the sample. If the cloth is stained the dyestuff is fit for use only in light or medium shades. Fastness to perspiration is best tested by a practical trial with the coöperation of workmen in the dyehouse. Dyestuffs that change shade upon ironing should be avoided as it consumes too much time to match colors under these conditions. No dyestuff is perfect. Dyes for goods of class (1) should be fast to perspiration, those for classes (2) and (3) fast to light. The widely advertized vat dyes which possess extreme fastness to all destructive agencies, lack in many cases the brilliancy demanded of certain shades. A table is given in which about 30 dyes are listed under their German names, Schultz No., and their equiv. American names. This table indicates their fastness to light, washing, perspiration, crocking, artificial light, ironing, and the particular class or classes of goods for which each dye is best adapted. It is of interest to note that at the close of 1920 only 11.6% of the dyestuffs used by the firm represented by the author were of foreign manufacture.

L. W. RIGGS

Bleaching of printed fabrics. ROBERT WEISS. Sealed note No. 1418, Sept. 11, 1903. *Bull. soc. ind. Mulhouse* 86, 406-7(1920).—Object: bleaching of fabrics after printing or after printing and steaming, or after printing, steaming and washing. Instead of the usual treatment with dil. chloride of lime the goods are passed in a vat which rolls through a dil. bath of KMnO_4 to which NaHCO_3 or borax is added to avoid the formation of caustic alkali. The bath is then treated with H_2SO_4 , which removes the oxide of Mn, after which the goods are washed and dried. Report on sealed note No. 1418 of Robert Weiss. TH. STRICKER. *Ibid* 407.—The process for this purpose is new, more energetic than the usual Cl treatment and successful in some cases.

L. W. RIGGS

Action of hydrogen peroxide on cellulose. R. HALLER. *Text.-Forschung* 2, 79-84 (1920).—Most vegetable textile fibers are disintegrated by the prolonged action of 80% H_2O_2 and eventually dissolve with formation of dextrose. A cellulose peroxide which reacts characteristically with KI is formed as intermediate product. The disintegration of the fibers is accelerated by certain cotton dyes and retarded by others. Cotton mordanted with Cr oxide is very rapidly broken down, while the simultaneous presence of Al has a retarding effect. It is possible that impregnation of laundry goods with alumina may protect them from injury from detergents contg. H_2O_2 .

J. S. C. I.

The degumming of ramie. ROBERT WEISS. Sealed note No. 1412 Aug. 14, 1903. *Bull. soc. ind. Mulhouse* 86, 404-5(1920); *Mon. sci.* 11, 68(1921).—The degumming bath should contain 2 kg. each of coconut, olive and cottonseed oils, 16 kg. of white oleate soap and 370 liters of water at 40-50°. Ramie, whether previously soaked in dil. mineral acid and rinsed or not, is placed in a vat basket, introduced into the vat and the latter closed before the bath is admitted. Circulation of the bath is maintained for 30 min., then the bath is allowed to stand overnight, the temp. is raised to 100°, the bath circulated 1 hr. and drawn off to be used again. The fiber is washed twice with NaOH soln. at 130°, then with mineral acid. To improve the flexibility the ramie is treated in a bath containing 1.5 kg. each of coconut and olive oils, 1.5 kg. glycerol, 6 of white soap and 800 liters of water. Report on sealed note No. 1412 of Robert Weiss. TH. STRICKER. *Bull. soc. ind. Mulhouse* 86, 405.

L. W. RIGGS

Kemps. HOWARD PRIESTMAN. *Textile Am.* 35, No. 4, 24-6(1921).—A discussion of the structure of kemps and kempy wool with special reference to Bowman's work. Particular mention is made of flat kemps. Several expts. which support the theory are given.

CHAS. E. MULLIN

Preservation of fish nets. HARDEN F. TAYLOR. *Bur. Fisheries, Document No. 898*, 35 pp.(1921).—The most important methods of preserving nets are tanning, tarring and creosoting. Recipes and full discussion are given. JEROME ALEXANDER

Dyeing of straw pulp (PRESS) 23. Analysis of sulfonated oils (BUMCKE) 27. Improved formulas for the calculation of inorganic impurities in sulfonated oils (HART) 27. Digestion of woody fibers with chlorine for the production of cellulose and textile fibers (WARNYTO, GIERISCH) 23. Estimation of hydrosulfites and sulfoxylates (HELVIG) 7. Purifying alkaline waste liquors from disintegration of cellulose (GER. pat. 322,461) 23.

The Cotton Textile Worker's Handbook. By INTERNATIONAL CORRESPONDENCE SCHOOLS. 2nd Ed. Scranton, Pa.: International Textbook Co. 367 pp. \$1. For review see *Proc. Am. Soc. Civil. Eng.* 47, 564(1921).

Dyeing with the use of cleavage products of protein. CARL BENNERT. Ger. 331,234 and 331,484, Dec. 25, 1915. (Additions to 330,133 (C. A. 15, 1080)). Animal or vegetable fibers can be dyed with vat dyes in weakly alk., neutral or more or less weakly acid vats provided cleavage products of proteins, such as protalbinic or lysalbinic acid or the like, or salts thereof in purified or unpurified state are added to the dye bath. The corresponding acids also of cheaper proteins, *e. g.*, of casein, or of rice or wheat gluten are suitable for the purpose, it being sufficient to purify the acid mixts. with $\text{Ca}(\text{OH})_2$ or $\text{Mg}(\text{OH})_2$. Cf. C. A. 14, 3159.

Dyeing with the use of cleavage products of proteins. CARL BENNERT. Ger. 332,476, Jan. 19, 1916. (Addition to 330,133 C. A. 15, 1080). The cleavage products mentioned in patent No. 331,234 (preceding abstract) may also be used in combination with the leuco compds., more or less capable of re-oxidation, of S dyes which in this state possess sufficient affinity for animal fiber. For the production of neutral leuco preps. free from caustic preps. of a certain alkalinity or acidity, the vat dyes produced as stated above may be mixed with acids, acid salts or neutral salts or organic acids containing volatile alkali.

Dyeing with aniline black. KARL SCHLATTER. Ger. 332,675, Mar. 23, 1919. The parts which are particularly subject to wear are made partly of crude thread which has been treated with a protective agent against the injurious action of the aniline-black process, or of thread dyed approx. black but not by the principle of aniline-black dyeing in agers. Or the raw material for the parts most subject to wear is printed, before being dyed with oxidation aniline-black, with a protective soln., or these parts or the yarns therefore, respectively, are treated, before being dyed black, with metallic oxide or metallic protoxide or other acid-binding or reducing substances or mercerized in a known manner. Or the parts most subject to wear may be made of two kinds of thread, one of which is either dyed with another dark color or, before being dyed with aniline black, pretreated with metallic oxide, metallic protoxide or other acid-binding or reducing substances, or mercerized.

Dyeing raw materials. CHEMISCHE FABRIK COSSWIG-ANHALT, G. M. B. H. Ger. 330,448, Oct. 29, 1914. When tannin dyestuffs are used, the preliminary bleaching process may be dispensed with, as stated in 288,687, (C. A. 10, 2528) the treatment with active O compds. being combined with tannin mordanting.

Bucking cotton goods in one operation. WALTER MATHEUSIUS and MORITZ FREIBERG. Ger. 323,992, July 15, 1916. The goods, in some cases pre-steamed, are de-aerated in old lye contg. about 80% of caustic alkali and purified by known means or in a mixt. of purified old lye and fresh lye, then bucked in fresh lye (3 l. per lb. of cotton) contg. 2.5-3% of NaOH relative to the total wt. of the cotton. The bucking

liquor is then displaced by weak soda or alkali lye, and the goods are finally washed hot and cold. The process permits considerable saving in hands and raw materials over the known bucking process. It requires 12-13 hrs.

Printing fabrics. CALICO PRINTERS' ASSOCIATION, LTD., F. ASHTON and G. NELSON. Brit. 159,552, Nov. 28, 1919. A process of printing textile fabrics comprizes the following steps in the order specified: (1) Printing with a wax, resin or the like resist; (2) immersion in a mordant soln. containing a Cr, Al, Fe or Cu mordant or mixts. of these; (3) drying, and stripping the resist in any suitable way; and (4) dyeing with a color appropriate to the mordant. The effect of mordanting the mechanically resisted fabric with the mordant specified is to fix a larger proportion of the mordant on the resist-printed fabric than on the unprinted part, with the result that when the resist is removed and the fabric subsequently dyed, the resist pattern is outlined in darker shades upon the colored background. Other operations may be superimposed upon the process. Thus, the fabric may be dyed with indigo or other suitable coloring matter between the first and second steps or between the second and third steps; or white cloth may be treated with a β -naphthol preparation prior to the first step, subsequently developed with a diazo soln. between the first and second steps; or the fabric may be printed with a discharge color, aged and washed between the third and fourth steps or the wax resist may be cracked for veined and marbled effects. Cf. 23,324, 1911 (C. A. 7, 1296).

Artificial silk and staple fiber. A. LAUFFS. Ger. 322,141, Dec. 3, 1918. The H_2O to be used for pptn. is freed of a part of its lime and the harmful action of the remaining lime is lessened by the addition of sol. org. compds. The removal of $Ca(HCO_3)_2$ may be effected by addition of soda, boiling, filtration in column app. or by other means. The purification is preferably accomplished merely by mechanical means, without addition of chemicals. Suitable org. compds. are different kinds of sugar, glycerol, sol. starch and the like. The Cu is removed more quickly and more thoroughly than without such additions. The resultant textile fibers are distinguished by a high degree of uniformity, especially in the dyeing process.

Artificial fibers from cellulose solutions. MRS. LUDWIG SCHREINER née SAPPES. Ger. 322,538, Dec. 25, 1917. Conc'd. $CuO-NH_3$ cellulose soln. and slowly acting gaseous precipitants are employed. The same gas is used continuously in a cycle, the NH_3 being abstracted from it after having been used and then employed anew. Atmospheric air freed, as far as practicable, of O, may be used as the gaseous precipitant. The conc'd. $CuO-NH_3$ cellulose soln. must be freed as far as possible of excess NH_3 . The spinning openings have a width of about 0.8 mm. and are arranged in the form of a rose-head about 7 mm. apart, in order that the thick drops which are likely to form in the beginning of the spinning operation may not touch one another. As soon as these drops are detached by their own wt., they will draw behind them an exceedingly fine thread and enter an acid bath in which the lower end of the spinning cylinder is immersed. There the threads are seized by a hook and placed on the reel in the shape of a bundle of fibers.

Textile fibers from plants. ERNS V. and ALFRED BESENBRUCH. Ger. 322,167, Nov. 8, 1918. The plant parts to be treated are first subjected to the action of vapors produced from acids or from alk. solns. of Na sulfide, bisulfites, bisulfates, peroxides, hypochlorites, permanganates, or the like, and then boiled in solns. of alkali, Na_2S or the like, contg. fats, oils or soaps. When for the first stage of the process solns. of Na sulfide, bisulfates or non-volatile acids are used, it should be noted that these substances even when they are brought up to the plant parts to be treated in the form of vapors, are entrained by the water bubbles of the wet steam, and that the action of the same is most effective at places where the fibers have just been opened out by the action of the steam and are, therefore, particularly susceptible to the action of chem-

icals. The boiled material is washed. Thereby the finer fibers are made to sep. from the coarser woody fibers. The former are carried off by the rinsing water and collected, while the woody fibers are in some cases boiled anew, rinsed and bleached.

Separation of the leaf and root parts from the stalk fiber in the preparation of textile fibers from peat material. AKTIESELSKAP NORSK TORVTEXTIL. Norw., 30,805, June 14, 1920. The mechanically cleaned fibrous material is treated for a few min. with a weakly acid liquor, which, while attacking the thin-walled leaf cells, will not attack the bast cells. The original toughness of the bast cells is then restored by treatment with weak soda lye, whereupon the mass is completely dried and finally broken. The bast cells will remain unimpaired, while the leaf parts, etc., are broken into small bits, which may be removed by sifting or screening.

Treatment of cellulose threads. PIERRE JOLIOT. Ger. 322,047, Apr. 24, 1914. The treatment is conducted *in vacuo*, whereupon the S is abstracted from the threads, after they have been converted under pressure into cellulose by the action of an alkali sulfide soln. The elimination of the sulfides is then effected by means of a soln. of mineral or organic acid. Example: The threads, after having first been converted into xanthate, regenerated under pressure and washed, are subjected to a second similar treatment and then freed of the impurities pptd. during the regenerating process. The threads thus obtained are stated to be more lustrous, more transparent and softer than those which have been subjected only to a single treatment. They are also claimed to take on dye-stuffs more readily than the latter.

Treating threads with liquids. HILAIRE OR CHARDONNET. Ger. 327,323, July 27, 1915. A process and machine are described for treating thread, more particularly artificial silk threads, by means of liquids, for the purpose of denitrating, bleaching, dyeing or washing the threads under the action of centrifugal force.

26—PAINTS, VARNISHES AND RESINS

A. H. SABIN

Surface tension and interfacial tension of varnishes and paint liquids. HENRY A. GARDNER AND P. CHARLES HOLOT. Paint Mfrs. Assoc. U. S., *Circ.* 124, 11 pp. (April 1921).—Measurements of surface tension of paint and varnish liquids against air by the DuNoy app. (*C. A.* 14, 1549) show that liquids of the same general type have such slightly different surface tensions that the measurements are of little assistance in explaining the great differences in wetting power, tendency to draw together in drops, etc. Tabulated results show about the following averages in dynes per cm.²: triglyceride drying oils, 38.5; turpentine, 31.5; mineral spirits, 30. Interfacial tension between liquid-liquid or liquid-solid surfaces is probably of far greater significance in the paint industry. The authors measure relative interfacial tension between immiscible liquids by counting the drops formed by a definite vol. of the liquid of lower sp. gr. when it is permitted to bubble up through a capillary immersed in the liquid of higher sp. gr. The app. consists of a 5 cc. pipet bent upward at the lower end and drawn to a point having an orifice of about 0.8 mm. After filling the pipet, the U-shaped end is immersed to a definite depth in a beaker of the liquid of higher sp. gr., and then air is admitted slowly enough so that the rising drops from the end of the pipet may be counted. The no. of drops is inversely proportional to the interfacial tension. These results on paint liquids against H₂O, vary widely; some of them are: raw linseed oil, 55 drops; perilla oil, 37 drops; linseed fatty acids, 300 drops; turpentine, 115 drops; mineral spirits, 79 drops. The high interfacial tensions of perilla and chia oils probably account for the "crawling" when flowed on glass. Addition of linseed fatty acids to raw linseed oil greatly decreases interfacial tension. Since all solid surfaces are be-

lieved to hold an adsorbed film of moisture, the liquid-liquid interfacial tension measurements made, may closely apply to the conditions obtained between pigment particles and vehicles in paints. The ratio between the no. of drops of turpentine to mineral spirits, measured in H_2O was approx. the same as in salt solns. Different vegetable oils, and all groups of liquids of a similar type, may exhibit equal surface tensions because they contain a common atomic group which is the least active portion of their molecules and which concentrates at the surface, as explained by Langmuir (*C. A.* 11, 2849); but their interfacial tension with another liquid or a solid may bring different atomic groups of the molecules to the interfacial surface, and therefore produce very different manifestations even though both effects are the result of tension forces.

F. A. WERTZ

Physical testing of paint and varnish; suggestions regarding possible methods and apparatus. HENRY A. GARDNER. *Paint Mfrs. Assoc. U. S., Circ.* 122, 13 pp. (April 1921).—Standard bond papers were coated with a wide variety of varnishes, dopes, and paints; and the coated sheets after drying and again after exposure, tested in the usual paper testing app. for thickness, bursting and tensile strength, folding endurance, etc. Interpretation of tabulated results must await further development of the test. G. has experimented with a modified Pfund cryptometer (*C. A.* 14, 637) to give direct reading of hiding power on ready mixed paints; and suggests the study of the effect of the color of undercoat on durability of clear finishing varnishes; and related problems.

F. A. WERTZ

Note on fire-resisting paints and varnishes. HENRY A. GARDNER. *Paint Mfrs. Assoc. U. S., Circ.* 121, 2 pp. (April 1921).—Duplicate sets of test shingles coated with "Pamak" fire-retardant paints (*C. A.* 14, 3802), and with the same paint to which 10% of $(NH_4)_2HPO_4$ -linseed oil paste had been added, were ignited and then withdrawn from the flame. The latter showed very slightly greater fire-retardant properties, but because of its H_2O -sol. constituent, it is not to be recommended except for interior use. "Phosphated" varnishes also might find some application although they give a rough film. Zn borate added to paints gives excellent exterior service and retards burning by producing a glass-like film under the action of heat which prevents escape of combustible gases from the wood.

F. A. WERTZ

Suggestions regarding metals for varnish kettles. HENRY A. GARDNER. *Paint Mfrs. Assoc. U. S., Circ.* 125, 3pp. (May 1921).—The losses in wt. of small metal strips of Al, steel, Cu, Monel, and Ni on suspending them in highly acid varnishes for 2 hrs. at 500° were detd., and the effect on color of the varnish was noted. Ni was found to be next to Al in resistance to acid and paleness of the resulting varnish, so that Ni varnish kettles appear to be desirable. Physical properties and analyses of the metals used are tabulated.

F. A. WERTZ

Comparison of durability and water resistance of varnishes. E. G. BLOCH. *Republic Varnish Co. Drugs, Oils, and Paints* 36, 426 (1921).—To det. whether the resistance of a varnish to turning white on immersion in water bears any relation to durability on exposure, B. tabulates exptl. results on so-called water proof varnishes and on auto finishing varnishes. These tests show that if a varnish does not turn permanently white when water gets on it, then it does not necessarily lack durability. Details of the prepn. of panels, etc. are given.

F. A. WERTZ

Lithopone—its manufacture and uses. A. S. KREBS. *Drugs, Oils, and Paints* 36, 428-9 (1921).—An address.

F. A. WERTZ

American tung oil exploration; climatic and cultural conditions, examinations of oils, etc. HENRY A. GARDNER. *Paint Mfrs. Assoc. U. S., Circ.* 123, 20 pp. (April, 1921); cf. *C. A.* 14, 3803.—G. reports on the inspection of the principal tung oil plantations in the South. Oils expressed from 100 lb. lots of tung oil fruits from different

communities, were all of a very light color, neutral, and in general of the very highest quality,—superior to the best importations from China. Complete data are tabulated.

F. A. WERTZ

The evaluation of white pigments with special reference to antimony oxide. H. E. CLARKE. *J. Oil Colour Chemists' Assoc.* 4, 2-26(1921).—C. gives basic definitions of the desirable properties of white pigments, the effect of fineness of pigment particle on oil absorption and hiding power of the pigment itself, and on the "galloneage" opacity of the paint produced from it, etc. Sb_2O_3 for pigment purposes is a fume product made much like ZnO and is marketed as "Timonox"; it is, however, a condensed oxide vapor instead of an oxidized metallic vapor like ZnO . Color is white to pale ivory; sp. gr. about 5.4; has a very soft texture, low oil absorption and requires about 10% oil for grinding to a stiff paste. The relative hiding power of "green star" is 85.4 and of "red star" 77.6 as compared with lithopone 100, and white Pb 57.6. The relative opacities of paints of approx. equal consistency ("galloneage" opacity) are: Timonox, 95.5-96; ZnO , 63; white Pb, 77. Exposure tests show it to give more durable paint films than white Pb. H_2S has only a slight yellowing effect which reaches a max. and bleaches out on exposure to sunlight. This is probably due to a progressive oxidation of the Sb_2S_3 formed, so that the degree of yellowing is dependent on the nature of the catalytic oxidizers in the oil, and the concn. of the H_2S . The pigment has no drying action on linseed oil, and even though sufficient driers have been added for proper drying in clear weather, damp weather may retard drying for 3-4 days, in which case the film invariably dries flat, sometimes showing wrinkle and crack formation. This might be due to hydrolysis of some Sb salt present, such as the sulfate, whereby an increase in the no. of pigment particles would result. Drying without a gloss does not seem to impair durability. Discussion of the paper brings out the fact that Timonox paint films have a tendency to soften, perhaps due to a reduction of linolein by the Sb_2O_3 . The addition of ZnO prevents this softening by neutralizing the liberated fatty acids. Timonox does not liver or "feed up" with any of the usual paint vehicles due perhaps to its acidic nature; this adapts it particularly for making enamels with tung oil varnishes. It has produced good results in printing inks.

F. A. WERTZ

A classification of pigments—with a descriptive outline of the manufacture of some dry colors. G. C. McEWEEN. *Can. Chem. Met.* 5, 108-10(1921).—Review.

F. A. WERTZ

Lead, manganese, and cobalt driers. WILLI FLATT. *Farben-Ztg.* 26, 1441(1921).—The relative strengths at 10° of the 3 best catalytic driers are 1 part Pb to 100 varnish, 20 hrs.; 1 part Mn to 400 varnish 20 hrs.; 1 part Co to 1200 varnish, 16 hrs. At 5° the drying time for the above mixts. is Pb, 24 hrs., Co, 36 hrs., Mn, 44 hrs. The drying action of Pb is least affected by temp. so that combinations of the metals are the most generally useful. An oil contg. Pb thickens and dries gradually and progressively while a Mn oil shows no evidence of drying until more than half of the required time has elapsed when the film suddenly loses its fluid character. Some Pb is, therefore, desirable in most driers.

F. A. WERTZ

Ostwald's color circle in practice. HANS WAGNER. *Farben-Ztg.* 26, 1505-7(1921).—For practical purposes, a color circle of 12 standard components usually gives a sufficiently accurate color designation. H_2O -sol. aniline dyes are best adapted for standards because they are easily prepd. and more nearly approach spectral colors in purity. A list of suitable dyes is given. (cf. C. A. 14, 2747.)

F. A. WERTZ

Stamping inks. WILLY HACKER. *Farben-Ztg.* 26, 1508-9(1921).—A collection of receipts for making rubber stamp inks, laundry marking inks, etc.

F. A. WERTZ

The acidity of ink and the influence of bottle glass upon ink. C. A. MYNCHILL. *Analyst* 46, 129-35(1921).—M. discusses the function of acidity, the amt. of added acid in ink, inks without added acid, and the effect of $FeSO_4$ on acidity. Strong acid (HCl ,

H₂SO₄, or (COOH)₂ is detd. by distn. of 10 cc. of ink after addition of neutral AcONa. Three distns. of 100 cc. each are necessary to get at least traces of AcOH. M. gives a method for detg. the total acidity by bleaching with H₂O₂ and titrating with N alkali with phenolphthalein indicator. A blank is run. The H₂O₂ method is applicable to logwood inks by boiling 5 cc. with 20 cc. H₂O₂ and 2 cc. N NaOH till transparent, cooling, dilg. with 5-6 times the amt. of water and titrating the excess alkali with standard H₂SO₄ using Congo red. A blank is run. The necessity of acidity control of the mfg. process is emphasized and the acidity of several com. inks listed. In alk. glass the reaction is retrogressive; the alkali is first liberated by water from Na silicate or Na₂CO₃, dissolves again, entering into combination with other constituents of the glass. In chem. work the chief difficulties are caused by soln. of Fe from the glass rather than by the alkalinity of the glass bottles. Discussion of the paper showed that the electrometric method of detg. total acidity is favored. The use of methyl red for titration of FeSO₄ solns. is suggested.

W. H. BOYNTON

Solidification of oils for the manufacture of linoleum. FELIX FRITZ. Berlin. *Kunststoffe* 11, 41-2(1921).—The various methods of oxidizing linoleic acid as practiced in the linoleum industry are discussed.

C. J. WEST

Solidification of oils. FELIX FRITZ. Berlin. *Kunststoffe* 11, 49-50(1921).—Various methods of thickening unsatd. oils are described and patent references given.

C. J. WEST

Turpentine oil or substitutes. W. GUSNER. *Seifensieder-Ztg.* 48, 191-2(1921).—A plea for the use of turpentine in leather cremes as against Lüddecke's advice to the contrary. (Cf. C. A. 15, 766).

P. ESCHER

Turpentine oil or substitutes. LÜDBECKE. BENSHKIM. *Seifensieder-Ztg.* 48, 339-41(1921).—A reply to Gusner's criticism (cf. preceding abstr.), insisting upon the advantages of adding substitutes, especially "decalin" (a new hydrogenation product of naphthalene) to turpentine oil for the use of shoe-cremes, and floor waxes. Results of distns. of various mixtures of decalin, light and heavy naphtha and turpentine are tabulated to show the relation of their b.p. to pure turpentine.

P. ESCHER

The acid constituents of pine resin: *d*- and *l*-pimaric acids. GEORGES DUPONT. *Compt. rend.* 172, 923-5(1921).—Crude pimaric acid was obtained by forming its Na salt from the resin of *Pinus maritima*, and crystg. it from H₂O. Recrystn. of this prepn. from AcOH then from EtOH gave *d*-pimaric acid $[\alpha]_D^{25} = 75.4^\circ$. During the AcOH crystn. racemization occurred, increasing the amt. of the *d* form. Pure *l*-pimaric acid, $[\alpha]_D^{25} = -282.4^\circ$, was obtained by recrystn. from aq. EtOH of varying concns. To avoid racemization, the temp. should not go above 60°. Also in *Bull. Soc. Chim.* 27, 718-27(1921).

T. G. PHILLIPS

Swedish rosin oil (HÜBSCHER) 22.

Paints. C. H. IVINSON and G. S. ROBERTS. *Brit.* 150,543, Oct. 28, 1919. Anti-corrosive paints, consisting of asphaltum dissolved in a suitable solvent with or without other ingredients, are colored by lime-proof pigments such as Fe₂O₃ or ZnO, which are ground to a paste in a vehicle such as turpentine or linseed oil and then added to the paint. Alternatively, the pigments may be ground in the solvent used in the manuf. of the paint.

Color mixtures for painting purposes. NICCOLO SILVESTRINI. *Ger.* 322,556, Dec. 21, 1912. To the color mixts. camphor is added, which may be further admixed with glycerol wax, ether and unboiled linseed oil. A good mixt. consists e. g., of 100 parts of distn. products of crude mineral oil of 0.64 to 0.722 sp. gr. (especially gasoline, benzine and ligroin), 1-80 parts of ether, 1-30 parts of camphor and 1-5 parts of wax. Colors of every kind are capable of being dissolved in the liquid thus formed. The re-

sulting paints can be applied to wood, cement, gypsum, stone or the like. The paints may also be used for art painting.

Production of a weather-proof color-binding agent. ERICH STEPHANI. Ger. 321,113, July 3, 1917. Coloring materials insol. in H_2O are mixed with emulsions of solid hydrocarbons. The solid hydrocarbon is preferably mixed with the soln. of an emulsifier wholly or partly removed, and a colloid which is suitable as color-binding agent for the particular employment of the emulsion added as emulsifying agent. Or, after removal of the emulsifying agent, the finely divided product which seps. out may be suspended in a liquid which does not perceptibly dissolve the hydrocarbon. The resultant products form whitish slimy masses, which are capable of being mixed with any pigment to obtain a product sufficiently liquid for painting. Example: A suitable binding agent is obtained by emulsifying 25 parts by wt. of paraffin in a soln. of 5 pts. by wt. of stearate of ammonium, washing out the soap with H_2O , and mixing the very finely divided paraffin sepd. out with a little casein dissolved in NH_3 .

Paint-binding agents from mineral oil. JOSEF SOUKUP. Ger. 321,823, Nov. 1, 1918. Vaseline oil is dehydrated, in certain cases after being purified, by boiling with slaked lime, then beaten with substances giving off O such as MnO_2 and PbO_2 , to 100–120°, and then boiled with 50% of resin until all moisture has been removed, whereupon the mixt. is left standing, dild. with benzene or oil of turpentine, and poured off from the sediment. The liquid is ready to be used as paint immediately or after addition of mineral color or the like. It is more or less viscous, of light to dark-brown color, and possesses good staining properties. When applied to wood and Fe, it dries within 20 hrs. forming a well adhering coating of high luster, which resists atm. influences, pressure and friction.

Silicate paints or paint carriers and barium chloride. CHR. AUG. BERINGER. Ger. 320,863, July 20, 1918. Pure or mixed silicates of Ba obtainable by calcining barite with silicic acid or clay are converted, with chlorides in aq. soln. or suspension, into the corresponding silicate and $BaCl_2$. Example; 100 kg. of Ba-Al silicate, obtained by calcining clay with barite, are converted in a stirring vat at 80° with 500 l. of $MgCl_2$ soln. The Mg-Al silicate formed is pressed off in a filter press from the dissolved $BaCl_2$. The final products obtained are $BaCl_2$ and a dough-like white, which is a valuable substitute for satin-white.

Ink. F. C. FITZGERALD. Brit. 159,809, Oct. 4, 1920. A lithographic ink avoiding necessity for damping the stone or other printing surface comprises ordinary lithographic ink incorporated with a mixt. of one or more grease-resisting materials, such as glycerol, glycol, levulose, or other polyhydric alcohols, or Mg butyrate or like esters, and one or more deliquescent materials such as $CaCl_2$ or $Ca(NO_3)_2$, K phosphates, Na lactate, or NaMg chloride. Other ingredients may be added, e. g. gum, cresol or phenol, organic acids such as oxalic or tartaric acid, soaps such as Na or K stearate or palmitate, solvents such as ether, alc., benzene, EtOAc, or CCl_4 , and thin lithographic varnish.

Art varnish. WILHELM SCHWARZ. Ger. 322,802, June 14, 1918. Low-grade coumarone resins are purified by means of H_2SO_4 , whereupon O or air under pressure is passed through the beaten mixt. for several hrs. The resinous mass is emulsified with H_2O at 70–100°, and in order to obviate the sepn. of resin and H_2O and to render the emulsion insol. in H_2O , a glue soln. mixed with $K_2Cr_2O_7$ is added to the emulsion.

Synthetic resins. K. KULAS and K. PAULING. Brit. 159,494, Dec. 1, 1920. In the production of resinous condensation products from phenol or its homologs and $HCHO$ or its polymers or $(CH_3)_4N_4$, the materials are first heated with an acid condensing agent in large excess. Fusible resins sol. in a variety of org. solvents are thereby obtained; if sufficient $HCHO$ has been used, the resins may be converted by further heating to insol. infusible products. Numerous examples are given.

Synthetic resins. BAKELITE GBS. and R. HESSEN. Brit. 150,461, Feb. 15, 1921. Resinous products are obtained by first condensing phenols with aldehydes in the presence of a basic condensing agent and then adding to the oily or resinous condensation product an acid or substance of acid reaction. If the acid is added only in amt. sufficient or less than sufficient to neutralize the base present, the product obtained is a "resole" i. e. a resin which can be converted into an insol. infusible condensation product ("resite") by further treatment; if, however, the acid is in excess of this amt. but insufficient to produce the "resite" then a non-hardening resin (novolake) is obtained. The acids used may be inorg. or org. and solid, liquid, or gaseous, e. g. HCl, H₂SO₄, SO₂, oxalic, citric, etc.; or alkali bisulfates may be used. Org. acids may be used in larger excess than mineral acids, without formation of the "resite." Suitable parent substances are phenol, cresol, etc., and HCHO or its polymers or (CH₂)₆N₄. Examples of the product on each of the two types of resin are given.

Resin-like condensation products. AKTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKATION. Ger. 332,334, June 28, 1918. HCHO or substances generating HCHO are made to act on halogeno substitution products of naphthalene in the presence of acids.

Resin-like condensation products. AKTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKATION. Ger. 332,391, Jan. 9, 1918. Metal halides with or without addition of solvent, are made to act on halogen addition products of naphthalene or of its derivs., or on halogen derivs. of hydrogenated naphthalenes, by themselves or mixed with aromatic hydrocarbons.

27—FATS, FATTY OILS AND SOAPS

E. SCHERUBEL

Progress in technical fat chemistry during 1919. W. FAHRION. *Chem. Umschau* 27, 238-42 (1920).—A brief résumé under the following heads: Production of fats; refining; hydrogenation; boiled oil and lacquer; linoleum; cleavage; glycerol; stearin and olein; soaps; waxes; resins; mineral oils; miscellaneous.

P. ESCHER

Wool fat. VII. The "oily acid." I. LIFSCHÜTZ. *Z. physiol. Chem.* 110, 29-40 (1920).—L'a. expts. indicate that the acid portion of wool fat contains no oleic acid or other unsatd. fatty acid. After repeated purification, the so-called "oily acid" gives no reaction for oleic acid with chromic and sulfuric acids, and the low I value previously ascribed to this "oily acid" probably depends on the presence of an acid formed by the oxidation either of cholesterol or, as the absorption spectrum indicates, of ischolesterol.

J. S. C. I.

Fat-recovery problems during the war. CH. GRÄNACHER. *Schweiz. Chem. Ztg.* 1921, 151-5, 177-81.—Fat recovery from plants and animals, the improving and better utilization of present fats and synthetic fats are discussed. The use of yeast as a source of fat was developed and a content of 25% obtained which yielded an edible fat of a compn. between that of olive and rape oil. The hydrogenation of oils received considerable attention and the converse process of making soft fats from hard fats was worked out. Fats of high m. p. are first split into glycerol and fatty acids and the fatty acids esterified with low-molecular alcs. like glycol or EtOH, whereby fatty acid glycol esters and fatty acid ethyl esters are obtained, which possess properties similar to fats. Great difficulty was experienced in the technical operation of this process. It was found necessary to use a catalyzer and to have the alc. present in excess; and it is interesting to note that the catalyzer giving the best results was the Twitchell reagent. Large quantities of rancid fats high in free fatty acids were converted into neutral fats by esterification with glycol. Two problems had to be solved: the synthetic manuf. of fatty acids and of glycerol. Many attempts were made to synthesize glycerol

from C_6H_6 , but none proved practical, whereas the hlochemical method using sugar was successful. The efforts to synthesize fatty acids were concerned with the naphthenes and satd. and unsatd. aliphatic hydrocarbons. The naphthenes are least adapted for making fatty-acid-like substances on account of their unpleasant odor. The unsatd. aliphatic hydrocarbons are found mainly in brown-coal-tar oils and are well adapted for making fatty acids by the addition of ozone according to the method of Harries. During the war the process was undertaken on a large scale but it took 150,000 tons of tar to make 6,000 tons of fatty acids so that the conversion of satd. aliphatic hydrocarbons offered better results. By using NO_2 as oxidizing agent yields of 100% fatty acids were obtained. A pure synthetic edible fat was made during the last years of the war and put on the market. It was a fatty acid glycol ester. F. SCHERUBEN.

Catalytic reduction of unsaturated fats. A. S. Koss. *Przemysl Chem.* 4, 39-48(1920).—The catalytic activity of the oxides of Ni in the hydrogenation of oils at 255-60° (cf. Bedford and Erdmann, C. A. 8, 833) depends on the method used to prep. them. Addition of NiO to the sesquioxide does not always increase the activity of the latter (cf. *loc. cit.*). The particular combination most suitable for the treatment of any given oil must be ascertained by expt. Ni_2O_3 loses its activity on heating in the air, even at 115°. Its activity is considerably increased by very small quantities of Pt and this fact may account for some of the results obtained by Bedford and Erdmann. The resistance of drying oils to catalytic reduction is due to the position of the double linkages in the linolenic acid mol. Those between the 9th and 10th and the 12th and 13th C atoms are readily reduced, while that between the 15th and 16th is only very slowly attacked. The partially reduced oil therefore remains liquid owing to the formation of a liquid oleic acid. Ordinary oleic acid is readily reduced and possesses no drying properties. The drying properties of linolic acid are due to the double linkage between the 12th and 13th C atoms. The further a double linkage is from the carboxyl group the more difficult is it to reduce and the greater is the tendency to polymerization. These differences in the behavior of double linkages are well shown by means of a weak catalyst like Ni_2O_3 . J. S. C. I.

Hardening of fatty oils supplement I and II. SEIICHI UENO. *J. Chem. Ind. (Japan)* 23, 1109-28, 1203-17(1920).—In supplement to a previous article (cf. C. A. 10, 535) U. records the effect of impure H_2 , hardening of low-grade raw oils, use of spent Ni, effect of temp., uses of hardened oils and theory of hydrogenation. H_2 contaminated with O_2 , or repeated use of the same H_2 , contg. hy-product gases gradually decreases the effectiveness of the catalyzer. Such gas can be purified by passing it through charcoal. H_2 when admixed with O_2 is not dangerous in the oil-hardening process, but is apt to cause explosion if used in the manuf. of the catalyzer; after the catalyzer is once mixed with the oil or hardened oil it is safe to use impure H_2 . Low-grade raw oils such as tanked herring oil are best hardened after treatment with kambara earth or fullers earth. When oil is old or contains large amts. of free acid and other impurities, it should be treated with alkali before refinement. The refining action of the acid earth is increased if a better grade of oil is added to the low-grade oils. Spent Ni catalyzer that has lost its activity can be used to refine raw oil for hydrogenation, as it seems to have power to absorb the poisons in the oil. The method of converting the spent Ni to an active catalyzer is described. Settling of Ni catalyzer can be best effected by use of magnetic force. U. records that freshly made hardened oil contained in a mold is electrically charged. Hydrogenation can proceed at comparatively high temp. (280°). Various uses of hardened oil and oil saponid. in an autoclave are discussed. Prepn. of hardened wax and fatty acids for purpose of candles is given in detail. According to U., glycerol is better purified with Zn dust than with charcoal. The theory of hydrogenation according to U. is as follows: Catalytic action of Ni is due to its adsorption of H_2 and oil. In the first stage, the heat of adsorption helps to start the catalytic reaction by giv-

ing a shock to the reacting mixt. Then adsorbed H_2 combines with glycerides adsorbed on the surface of the catalyzer. As soon as the oil is hydrogenated, it is freed from the catalyzer. The velocity of adsorption of fatty acids and glycerides depends upon the degree of unsat. Anti-catalyzers are those substances which injure the surface of the catalyzer, weakening the absorption power. When a poison is present in an oil, the catalyzer adsorbs or absorbs it from the oil and its catalytic action is retarded. The refining action of the acid earth is due to the adsorption or absorption of the poison from the oil by the acid earth before the catalyzer can adsorb the poison. S. T.

Vegetable oils. H. D. TREFFT. *Can. Chem. Met.* 5, 102-4 (1921).—Discussion of recent developments in the edible-oil industry. E. SCHERUBEL

Brazilian oil palms. E. DE WILORMAN. *Mat. grasses*, 13, 5777-80 (1921).—The best known Brazilian oil palm is called the Uanassu. Its copra contains H_2O 4.21%, oil 66.12%, albuminoids 7.18%, carbohydrates 14.47%, cellulose 5.99%, and ash 2.53%. The oil has the following consts.: solidifying point 72.8° , sapon. no. 246.7, no. 36.9, glycerol 13.2%, R. M. no. 6.2, Polenske no. 11.3. The cake resulting from the extrn. of the oil contains H_2O 11.59%, oil 6.5%, albuminoids 19.81%, carbohydrates 40%, cellulose 16.5% and ash 5.60%. The oil is similar to coconut oil and in general can be used for the same purposes. E. SCHERUBEL

Cultivation of the African oil palm. ANON. *Bull. Imp. Inst.* 18, 209-52 (1920).—A descriptive article designed to supply information which will be of service to planters contemplating the establishment of oil-palm plantations. The palms give a big yield of pulp (62% of the wt. of the fruit) and a low yield of kernels (8%). The fresh pulp of the fruits contains about 55% of oil and the kernels contain about 50%. R. L. SIBLEY

Cottonseed and cottonseed oil. F. C. C. ROBB. *Pharm. J.* 105, 400-1 (1920).—A brief review of its technology. S. WALDBOTT

The analysis of sulfonated oils. An improvement of the Hart method. C. GERHART BUMCKE. *J. Am. Leather Chemists Assoc.* 16, 7-8 (1921).—B. finds that the Hart method for combined SO_2 using 25 cc. of 0.5 N H_2SO_4 gives lower results than are obtained by the wet method or the ash method. He gives results which show that if the strength of acid be increased to normal and 25 cc. of N acid be used and the soln. thoroughly boiled, that good results will be obtained. J. S. ROGERS

Improved formulas for the calculation of inorganic impurities in sulfonated oils. RALPH HART. *J. Am. Leather Chemists Assoc.* 16, 159-61 (1921); cf. C. A. 14, 2866. —In the original formula, inorg. impurities = ash — $[0.0634 (2A + As) + 0.0473 (2A - As)]$ (1) As = organically combined SO_2 , expressed as mg. KOH per g. sample, and A = fixed alkalinity expressed in the same terms. This has been simplified as follows: Case one—When $2A$ is equal to or less than As it is evident that there can be no carbonate in the ash, and, therefore, the carbonate term, $+ 0.0473 (2A - As)$, is equal to zero, and the formula becomes — Inorg. Impurities = Ash — $0.0634 (2A + As)$ (2) Case Two—When $2A$ is greater than As , Na_2CO_3 will be found in the ash and the carbonate term must be retained. This gives the equation; Inorganic Impurities = Ash — $(0.0795 As + 0.0946 A)$ (3) Substituting in formulas 2 and 3 the % combined SO_2 for its equiv. As , and letting S = % combined SO_2 , we have $As = \frac{111}{100} \times 10 S = 14 S$. Making the substitution, we obtain in case one—When A is greater than $7 S$, that is in the absence of carbonates; Inorg. Impurities = Ash — $0.1268 (7 S + A)$ (4) And in case two; Where A is greater than $7 S$, that is in the presence of carbonates, Inorg. Impurities = Ash — $0.0946 (11.77 S + A)$. J. S. ROGERS

A revolution in the sulfur-oil industry? A. KUCKHOFF. Trani, Italy. *Seifensieder-Ztg.* 48, 310-1 (1921).—"Sulfur-oil" is a greenish olive oil extrd. from press cakes by CS_2 . The proximity to city dwellings of a new factory built by the Unione Industriale Olearia at Trani, Italy, forced the abandonment of the customary CS_2 as extrn.

medium, and trichloroethylene was substituted with marked success, diminishing the danger of fire and explosion, decreasing loss by corrosion, improving the color, odor and quality of the extd. oil, lessening the cost of insurance, etc., but increasing by $1/4$ the cost of the extn. medium.

P. ESCHER

What is meant by degree of cleavage? E. LUBLINSKI. *Seifensieder-Ztg.* 48, 230 (1921).—The soap-maker desires to know how much Na_2CO_3 is required to neutralize the free acids on the one hand, and on the other hand, if faulty calcn. shows erroneously a high cleavage, the latter may be arrested too early and cause a loss. The proper meaning of degree of cleavage is therefore the % of free acids referred to the saponifiable matter in the material to be hydrolyzed as expressed by the formula: $(100 \times \text{acid no.})/\text{neutralization no. of sepd. acids}$.

P. ESCHER

What is meant by degree of cleavage? H. KEUTGEN. *Marburg. Seifensieder-Ztg.* 48, 204 (1921).—In practice the sapon. no. of the fat is detd. before cleavage, and during the process the acid no is detd. and sometimes the sapon no. in addition. If the sapon. no. before cleavage is 195 and the acid no. after cleavage is 180, the degree of cleavage is $(100 \times 180)/195 = 92.3\%$. It represents the % free acids in the final product referred to the saponifiable matter in the original material.

P. ESCHER

What is meant by degree of cleavage? E. LUBLINSKI. *Zerbst. Seifensieder-Ztg.* 48, 337-8 (1921).—A criticism of F. Krist's views. (Cf. 2 preceding abstracts and C. A. 15, 2005).

P. ESCHER

Glycerol manufacture. VI. PAUL VERBEEK. *Seifensieder-Ztg.* 48, 64-7, 81-3, 103-5, 122-3 (1921); cf. C. A. 14, 3331.—Calc. of the diln.-ratio of glycerol and water.—Detailed directions are given for calcg. the diln. of glycerol by wt. according to the formula $x = 100(a-b)/b$, in which a represents the % glycerol of the starting material and b the % glycerol of the desired concn. Dilg. glycerol by vol. is calcd. according to the formula $y = 100[(a-b)/b] D_1^t$, x and y representing parts of water (by wt. and vol.) to be added. A hitherto unpublished sp. gr. table for glycerol is given, prepd. by Bein of the German "Reichsanstalt für Mass und Gewicht" at Charlottenburg, recalculated from Nicol's d_{20}^{20} and Gerlach's d_{15}^{15} table:

GLYCEROL TABLE d_4^{20} .

% Glycerol.	Nicol's.	Bein's.	Mean.
100.....	1.2609	1.2608	1.2608
90.....	1.2347	1.2355	1.2351
80.....	1.2077	1.2087	1.2082
70.....	1.1806	1.1808	1.1807
60.....	1.1534	1.1531	1.1532
50.....	1.1261	1.1254	1.1257
40.....	1.0991	1.0988	1.0989
30.....	1.0727	1.0723	1.0724
20.....	1.0469	1.0465	1.0467
10.....	1.0221	1.0224	1.0223
0.....	.9982	.9982	.9982

To allow for the contraction taking place on dilg. glycerol by H_2O the following equation is derived: $K = 100 - [(V(D_1^t - 1) + 100)/d_1^t]$ where K represents the % contraction based on the final vol., V being the vol. of the starting glycerol, D_1^t its density and d_1^t the density of the desired concn. Corrections for difference in temp. are calcd. with the help of a table which includes correction for the expansion of glass. The refining of glycerol. Some glycerols retain a greenish tint even after double distn. or treatment by bleaching carbon—. They are whitened by adding the complementary color

of violet, blue or red, the glycerol losing thereby its brilliancy; in time the greenish tint returns, owing to the instability of the dyes in such high diln. An approx. idea of the coloring power of the dyes is given by the "Diln. limits" which are for methyl violet 1:200,000, fuchsin 1:120,000, alkali blue 1:10,000 and safranin 1:100,000. These colors may be detd. in glycerol by filtering a larger amt. of it hot through white filter paper which retains and shows the added colors.

P. ESCHER

The hydrolytic alkalinity of pure and commercial soaps. R. G. SARAIYA. *J. Soc. Chem. Ind.* 40, 74T(1921).—S. points out an error in the paper of the above title (*C. A.* 15, 1825). T. R. BOLAM. *Ibid* 40, 74T. Reply to Saraiya's criticism.

JEROME ALEXANDER

Old German settled soap and "Oberschalseife." A. Z. *Seifensieder-Ztg.* 48, 223-5, 243-4(1921).—A detailed account is given of the old small-scale method of boiling soap from tallow and lye from wood-ashes; also of the early method of making settled soda soap, "almond" soap and "top-layer-soap" ("Oberschalseife"). The almond soap is a settled soap from soft fats and palm-kernel oil, with distinctive mottled appearance, produced by drawing an iron rod lengthwise and crosswise at short interspaces through the soap in the frame. Oberschalseife was originally only the hard top-layer of a boiled soap, but later the whole kettle contents were transformed into it by rapid cooling in special, small frames. Palm oil was used almost exclusively for the latter. P. ESCHER

Commercial soaps and washing compounds. H. KÜHL. *Kiel. Seifensieder-Ztg.* 48, 201-2(1921).—Washing compds. of colloidal kaolin and artificially prepd. Mg silicates possess a distinct cleansing property for certain technical uses. Addition of protective colloids like gelatin and NH_4 increases the colloidal condition. Bleaching compds. which liberate O like perborates should on account of their gradual decompn. have the date marked on the package when the guaranty expires. Soap powders should contain at least 10% fatty acids, and addition of 3% of soap to cleansing compds. other than soap powders is advisable.

P. ESCHER

Ultramicroscopic structure of soaps (DARKE, *et al.*) 2. Swedish rosin oil (HÜSCHER) 22. Aromatizing edible oil and fat (Norw. pat. 30,855) 12.

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Oils and fats. E. R. BOLTON and E. J. LUSH. *Brit.* 159,587, Nov. 23, 1919. The free fatty acids contained in oils or fats are neutralized by heating the oil or fat *in vacuo*

or in a current of neutral gas with the equiv. amt. of the mono- or a mixt of the mono- and di-glycerides of the fatty acids.

Deodorizing fats and oils. WALTER ALEXANDER. Ger. 322,347, April 4, 1919. The oil or fat is made to trickle through a receptacle charged with filling bodies, while steam passes through the receptacle in the opposite direction. At the bottom of the vessel is a discharge tube with U-shaped liquid seal, to which tube a device after the manner of a Mariotte's bottle is connected for the purpose of sepg. the oil from the simultaneously collecting H_2O of condensation. The treatment occupies only 2-5 hrs. while the process heretofore employed requires 8-20 hrs.

Rendering hardened whale fat suitable for the preparation of edible fat. COURT GROSS. Norw. 30,749, May 25, 1920. The whale fat is treated in centrifugal apps. at a temp. at which more or less of the most difficultly fusible glycerides of the fat which are sought to be eliminated are sepd. off in solid form, while the more readily fusible glycerides are present in liquid state.

Fungus cells rich in fat. REICHAUSSCHUSS FÜR PFLANZLICHE UND TIERISCHE ÖLE UND FETTE, G.m.b.H. Ger. 320,560, Sep. 2, 1917. During culture in nutrient solns. rich in sugar but poor in N, the seed cells and their progeny are kept at a certain distance from one another equal to about the sowing of up to 20 g. of moist pressed fungus material per l., so that any great accumulations of cells are obviated. Solid surface structures moistened with the nutrient soln. and supplied with air may be employed. It is claimed that in these conditions the fat content will increase to 25-30%. Cf. C. A. 14, 3810.

Catalyst for hydrogenation of fatty acids and their glycerides. ÖLVERWERTUNG, G.m.b.H. Ger. 330,811, Aug. 22, 1912. (Addition to 292,649, C. A. 11, 1915). A mixt. of NiO with a fat or fatty acid is heated in a H current, and the resulting colloidal distributed product is used as catalytic material for hydrogenation.

28—SUGAR, STARCH AND GUMS

F. W. ZERBAN

Report on sugar (for 1919). A. H. BRYAN. *J. Assoc. Official Agr. Chemists* 4, 321-9(1921).—No extended work could be done on the recommendations made in the previous report (C. A. 14, 1057), but a few expts. were made on some of Horne's recommendations. It was found that (1) mixing raw sugar samples on a glass plate and mixing them in a mortar gave practically identical results. (2) The quantity of Pb subacetate to be used in each case must be left to the judgment of the analyst. The same is true in regard to (3), method of reading the polariscope; B. has obtained more concordant results by averaging the readings than by Horne's procedure. Over 4000 tests on (4) have shown that the temp. correction formula proposed by Browne (C. A. 3, 2636) gives results correct to within 0.05° in 48.9% of all cases, and that the av. deviation from the actual polarization at 20° is within 0.01°. (5) The Baumé scale should be abolished, but in case of real need that advocated by the A. O. A. C. should be used. The method of standardising quartz plates, employed by the Bureau of Standards, on the basis of the new value established by Bates and Jackson (C. A. 6, 3538), will lead to erroneous readings in saccharimeters equipped with the German scale, and such plates should be standardized according to the Herzfeld-Schönrock value. Browne's suggestion of an American-made polariscope (C. A. 12, 2701), and particularly of the 20 g. normal wt. invites further study and discussion.—The following recommendations are made: (1) Recommendations (1), (2) and (4) of the 1916 report should be referred to the Carbohydrate Laboratory of the Bureau of Chemistry. (2) Methods of detg. Cu by reduction of the oxide in alc. vapors be further studied. (3) That the following method of prepg. raw sugar samples for analysis be adopted: Mix thoroughly on a glass plate in the

shortest possible time with spatula and glass or iron rolling pin in case of lumps, or in a large clean dry mortar. (4) That Browne's formula for correcting the polarization of raw cane sugars to the temp. of 20° be adopted; where the levulose is actually detd. the following formula should be used; $P^{20} = P^t + 0.0003^\circ S (t-20) - 0.00812^\circ L (t-20)$. (5) That the question of the adoption of the Baumé scale of the Bureau of Standards in place of the one now in use be further considered, possibly by a committee. (6) That where standard quartz plates for the German or Ventzke scale are sent for certification, the Bureau of Standards be requested to certify on the old value of 100° V. = 34.657 circular degrees. (7) That the question of an international normal sugar wt. be referred to a committee, and that the views of all members of the A. O. A. C. be ascertained.

F. W. ZERBAN

Densimetric and polariscopic standardization in reference to the associate referee's report on sugar. FREDERICK BATES AND R. F. JACKSON. *J. Assoc. Official Agr. Chemists* 4, 330-3(1921); cf. preceding abstr.—Anticipating Horne's suggestion in regard to the Baumé scale, the Bureau of Standards has prepd. a scale based on the sp. gr. values of Plato, on the temp. of 20°, and on the modulus 145. It has already come in to general use, and its adoption by the A. O. A. C. is recommended.—Objections are raised against the adoption of the 20 g. normal wt. (cf. *C. A.* 13, 1264).—Bryan's criticism of the Bureau of Standards' method of standardizing quartz plates is unfounded. All that an analyst has to do to get correct readings is to apply the correction found from the value of the control plate proportionately over the entire scale. No proof has as yet been furnished that B. and J.'s value of the 100 point is in error. Bryan's recommendation in regard to this matter should therefore be rejected.

F. W. ZERBAN.

The international saccharimetric normal weight question. ANON. *Intern. Sugar J.* 23, 192-3(1921).—An analysis of the replies to the questionnaire sent out by the British committee (*C. A.* 14, 3165) discloses that 96% of the chemists are using the half-shadow quartz compensating instrument. The normal wt. used by 70% is 26 g. in 100 metric cc., that by the remainder 26.048 g. in 100 Mohr cc. 72% are in favor of retaining the 26 g. wt. per 100 metric cc., on the grounds that a change would be inconvenient, expensive, and confusing, and that the relative error would be increased by the smaller wt. The other 28% prefer the 20 g. wt., for the reasons advanced by Browne (*C. A.* 12, 2701). No one urged the adoption of the Freuch normal wt. One chemist advocated 25 g., in case any change should be made, as this would combine the advantage of a smaller percentage error with the ease of weighing and calcul. claimed for the 20 g. wt.

F. W. ZERBAN

Essentials in the production of sugar cane. C. W. HINES. *Philippine Agr. Review* 11, 4; *Intern. Sugar J.* 23, 195-9(1921).—A general treatise on relations between soil nature, plant production, and fertilization, with particular reference to sugar cane.

F. W. ZERBAN

Sugar cane work in Queensland; report for 1920. H. T. EASTERBY. *Intern. Sugar J.* 23, 199-201(1921); cf. *C. A.* 14, 2870.—This report, issued by the Bur. of Sugar Expt. Stations, relates that const. progress is being made in the amt. of sugar recovered from the cane, reaching 12.89% in 1920. Expts. on cultivation, fertilization, and varieties have been continued. Good results are being obtained with the Java canes EK 1, EK 28, H 100, M 168, also from the Indian cane Shajahanpur No. 10, and with the Yuba cane.

F. W. ZERBAN

Inversion of cane sugar by silicic acid. ALBERT AND ALEX. MARY. *Centr. Zuckerind.* 1919, 420; *Z. Zuckerind. Tschoslov. Rep.* 44, 166(1920).—Cane sugar is inverted by colloidal silicic acid in the same manner as by acids sol. in water. The rapidity of the inversion with silicic acid increases with rising temps. and greater colloidal dispersion. Maximum inversion occurs between 54° and 63°. At temps. above this point silicic acid coagulates very readily.

JOHN M. KRNO

Refinery practice in beet sugar manufacture. WALLACE MONTGOMERY. *Chem. Met. Eng.* 24, 469-72(1921).—This is a general article describing modern practice in the manuf. of sugar from beet juice, showing the yield and compn. of the various products and giving formulas and a chart used for calcg. yields. Illustrated. F. W. ZERBAN

Extraction of juice from the sugar beet. WALLACE MONTGOMERY. *Chem. Met. Eng.* 24, 435-7(1921).—Modern methods of fluming, washing, slicing and diffusion are described, and the effect of various factors on quantity and compn. of the different products is pointed out. Illustrated. F. W. ZERBAN

The determination of ash in cane sirups and molasses. F. W. ZERBAN. *J. Assoc. Official Agr. Chem.* 4, 444-51(1921).—Coöperative work on the detn. of ash in 3 cane products, a sirup, a first molasses, and a final molasses, by the 3 official methods of the A. O. A. C. has shown that the direct ash method with the use of $(\text{NH}_4)_2\text{CO}_3$, and the direct ash method with sep. treatment of the sol. ash, again using after-treatment with $(\text{NH}_4)_2\text{CO}_3$, give the lowest av. probable error in the hands of different analysts, and also agree closely with each other. The correction factor of 10% used in connection with the sulfated ash method is much too low. In the products analyzed the correction was found to vary from 16.6 to 21.6 averaging 19%. The factor also varies with the amt. of H_2SO_4 used for carbonization. From the standpoint of close agreement between different analysts the sulfated ash method has no advantage over the direct ash method, and with most cane products it even has no advantage in case of manipulation. The continuance of the 10% factor may further lead to difficulties in the enforcement of the present official ash standards for cane products. It is recommended (1) that a study be made of the influence of different and known temps. of incineration on the results of ash detns. in cane sirups and molasses, carrying out the incineration in both Pt and silica dishes for comparison; (2) that a large number of samples of different grades of cane sirups and molasses be used for comparing ash detns. by the sulfate and direct methods to det., if possible, the proper correction factor to be applied.

F. W. ZERBAN

The determination of alkalinity. FRANT. JANÁK. *Oesterr. ung. Z. Zuckerind.* 1918, 440; *Z. Zuckerind. Tschoslovak. Rep.* 44, 282(1920).—A discussion of the possible use of the electrolytic measurement of the H-ion concn. for the detn. of the alkalinity or acidity of sugar juices. Its application as a control of satn. is pointed out. JOHN M. KRNO

Determination of alkalinity of raw sugars. G. BRUHNS. *Centr. Zuckerind.* 28, 935-6(1920).—Many raw (beet) sugars, if covered with water contg. red phenolphthalein give at first a yellow soln. which becomes red after some time owing to the gradual solution of Ca carbonate contained in the sugar. This behavior is sometimes shown by juices, sirups, and white sugar. The red color may eventually vanish owing to absorption of CO_2 from the air. In testing the reaction of sugar which is to be stored, such delayed reddening should be neglected, i. e., the sugar should not be returned as alk. for solid Ca carbonate affords little protection against acidification and deterioration during storage. If, however, the sugar is to be inverted for the production of sirup or artificial honey, any Ca carbonate present should be reckoned as alkalinity in view of the very small quantity of acid used for inversion. In such cases the alkalinity may be detd. by dissolving 20 g. of the sugar in 100 cc. of water and 5 cc. of 0.1N HCl, boiling for 5 mins., cooling and titrating back with NaOH. J. S. C. I.

Loss of alkalinity in stored raw sugar. FRANT. JANÁK. *Oesterr. ung. Z. Zuckerind.* 1919, 438; *Z. Zuckerind. Tschoslovak. Rep.* 44, 248(1920).—J. suggests that due to the action of higher temps., humidity and the catalytic action of carbonates and bicarbonates, free NH_3 is changed to HNO_3 which causes a decrease in the alkalinity. Raw sugars made from beets rich in N should therefore be stored in very dry warehouses and at the lowest temp. possible. JOHN M. KRNO

Extraction of beet juice by the "rapid" method. S. THEILER. *Centr. Zuckerind.*

28, 468-70, 492-5, 522-4(1920).—Expts. have been made in several factories in Germany with the so-called "rapid" method, in which after preheating by Bosse's scalding process (U. S. pat. 1,005,931; C. A. 6, 167) the slices are extd. by means of Paschen's inclined trough app. (U. S. pat. 1,134,152; C. A. 9, 1407), the juice and slices being forced from one end to the other in counter-current through a series of compartments. In the B&K factory, in which a trough about 22 m. long, divided into 20 compartments, was used, the sugar content of the treated slices was found to be 0.4% (calcd. on the wt. of sliced roots); but the d. of the juice finally drawn off, which amounted to 140% on the roots, was about 3° Brix less than in the case of the liquid ext. by the diffusion method. The purity of the juice was about the same as in the diffusion process; but the juice obtained by the new process was particularly low in coagulable substances. J. S. C. I.

Heat economy in the beet sugar industry. H. CLAASSEN. *Z. Ver. dest. Ing.* 65, 387-8(1921).—The heat economy in the boiler house does not differ materially from that in other industries, except that the evapg. station furnishes sufficient hot, pure water for boiler feed, and that the flue gases, after suitable purification, may be utilized for drying exhausted cosettes. About 30% of the fuel value of the coal is lost in the boiler house. The theoretical steam requirements of the sugar house itself are very high, but through the proper utilization of exhaust steam and of the vapors in the evapg. system they are reduced about 70%. Actually 60-65 kg. of steam are used per 100 kg. beets, including all ordinary losses. It has been proposed to reduce steam consumption by using high pressure steam evapg., but the advantages claimed have not been realized. Neither has the compression of the vapors from evaporators given practical results. Economies may best be effected by reducing radiation losses through shortening and insulation of pipe lines, centralization of machinery near the boiler house, more extended use of electricity and similar measures. A complete heat balance should be struck at regular intervals, enabling the engineer to detect and remedy avoidable losses. An example of such a balance sheet is given. F. W. ZIEBAN

The recovery of ammonia as a by-product in the sugar industry. ANT. RUZFF. *Z. Zuckerind. Tschoslovak. Rep.* 44, 239-43, 249-52, 257-9(1920).—An extended résumé of the various methods proposed for the recovery of NH_3 produced during the different stages of sugar manuf. and a discussion of the reasons for their failure when applied commercially. JOHN M. KRNO

The recovery of ammonia from the gases escaping during saturation considered from a physical chemical standpoint. ANT. RUZFF. *Z. Zuckerind. Tschoslovak. Rep.* 44, 377-81(1920).—R. advances several criticisms of the present method of recovering the NH_3 from the 1st satn. and from the NH_3 conduit of the 2nd evaporator unit as practiced in the refinery at Pečky. H_2SO_4 is used as an absorbing agent. Under the existing conditions the H_2SO_4 absorbs considerable amts. of H_2O and consequently the H_2SO_4 soln. could not be satd. with NH_3 to such an extent as to obtain crystn. of $(\text{NH}_4)_2\text{SO}_4$. The process also entails a considerable heat loss. R. suggests that the excess CO_2 , escaping in considerable amts. during satn., be utilized for fixing the NH_3 . $(\text{NH}_4)_2\text{CO}_3$ will be the first product formed from the reacting gases. Physico-chem. conditions, however, can be so adjusted that the more stable NH_4HCO_3 is produced. Consideration of Guldberg and Waage's law of mass action, Le Chatelier's theorem and the application of van't Hoff's equation to the reaction represented by $\text{NH}_3 + \text{H}_2\text{O} + \text{CO}_2 \rightleftharpoons \text{NH}_4\text{HCO}_3$ show that high pressure, low temp., and excess amts. of CO_2 and H_2O aid the formation of NH_4HCO_3 . The use of CO_2 in place of H_2SO_4 would be much more economical. JOHN M. KRNO

Destruction of sucrose during the boiling of low-grade massecuites. H. L. WHITT. *Intern. Sugar J.* 22, 588(1920).—See C. A. 14, 862. F. W. ZIEBAN

Loss of sucrose by inversion during the heating of second massecuites and final

molasses. R. C. PITCAIRN. *Intern. Sugar J.* 22, 589(1920).—See C. A. 14, 1233.

F. W. ZERBAN

Evaporation versus fractional freezing. ALB LINSBAUER. *Z. Zuckerind. tschosslovak. Rep.* 44, 159-60(1920).—A discussion.

JOHN M. KRNO

Evaporation versus fractional freezing. K. ANDRIK. *Z. Zuckerind. tschosslovak. Rep.* 44, 307-8(1920).—An answer to the above articles.

JOHN M. KRNO

Frothing in the raw juice preheaters and its prevention. ASKAN MÜLLER. *Z. Zuckerind. tschosslovak. Rep.* 44, 199-200(1920); cf. C. A. 15, 773.—Rapid circulation of the juice must be maintained by either pumps or turbines. Various patents are enumerated which cover the proper equipment.

JOHN M. KRNO

[Sugar] boiling scheme used in Java. G. E. G. VON STIETZ. *Louisiana Planter*, 65, 92-3(1920).—Good results are stated to be obtained by boiling three strikes, as follows: (1) Evaporator sirup mixed with second and third strike sugars, the purity being 85-90°; (2) some of the molasses from the first strike, the purity being 70-5°; (3) molasses from the second strike, together with the rest of the molasses from the first strike, the purity being 60°. Advantages of this method are that all the merchantable sugar originates from the first strike, giving a good and regular quality, and that in this first strike boiling is particularly easy, since it is not necessary to granulate, but only to evap. the mixt. of sirup and molasses sugar to a sufficient degree, and take in more sirup until the pan is full and until the massecuite can be transferred to the crystallizers. A waste molasses of 30-2° is finally obtained.

J. S. C. I.

A modern boiler house for the sugar factory. KAREL MÁZEL. *Z. Zuckerind. tschosslovak. Rep.* 44, 275-81(1920).—An address. A description of various types of boilers, automatic stokers and other equipment is given.

JOHN M. KRNO

A sugar house brixometer. ANON. *Intern. Sugar J.* 23, 208-9(1921).—The instrument designed by Deerr (C. A. 8, 2632) has recently been placed on the market. A description is given, with illustration, also a temp. correction table.

F. W. ZERBAN

Decolorizing carbons. STUART M. LITTLEMORE. *Chem. Eng. Mining Rev.* 13, 211-4(1921).—This is a review of patent and technical literature on the subject, especially of recent work by Chaney (C. A. 13, 3238) and by Zerban (C. A. 13, 82, 797, 1260, 2295).

F. W. ZERBAN

Preparation of maltose [from starch]. M. FALCH. *Z. ges. Brauw.* 43, 281-3, 289-91, 297-9, 306-9(1920).—The following method gives a yield of 30 pts. of pure maltose per 100 pts. of air-dry starch:—300 g. of potato starch is mixed with 1 l. of water and 30 cc. of malt ext. (made by digesting 1 pt. of pale kilned malt or green malt with 4 pts. of water for 3 hrs.), and the mixt. is poured quickly into 2 l. of boiling water, with stirring, whereupon the starch is rapidly liquefied. After the temp. has fallen to 60°, 30 cc. of malt ext. is added and the temp. is maintained for 2 hrs. after the liquid has ceased to react with I. The liquid is then heated to boiling, filtered, diluted to 7° Balling (sp. gr. 1.028), treated with 240 cc. of malt ext. and with 7 mg. of HF per 100 cc., and maintained at 30° in a stoppered vessel for 3 days, during which no turbidity should appear. After addition of Ca carbonate the liquid is filtered, heated to boiling, evapd. to a water-content of 30%, preferably *in vacuo*, and then seeded with maltose crystals and maintained at 40°. If crystn. is very slow 3 sep. quantities of 70 cc. of 90% alc. are added at intervals of 1 day. The crystals are collected with the aid of a pump, then mixed with 90% alc. and again collected. 100 g. of the pressed product is dissolved in 30 cc. of water on a water-bath, mixed with 260 cc. of 90% alc. heated to boiling, and filtered. After cooling and seeding with maltose crystals, the liquid deposits pure maltose.

J. S. C. I.

- Test code for evaporating apparatus (ANON.) 1. Report on maple products (SNELL)
12. The production of several organic acids from sugar solutions (MAZZADROLI) 16.

HERIOT, T. H. P.: *Rohr- und Rübenzucker-Fabrikation*. London: Longmans Green & Co. 411 pp. 24 s. For review see *Deut. Zuckerind.* 46, 135(1921).

MUNERATI, OTTAVIO: *Observations and Investigations on the Sugar Beet* (in Italian). Part I. Rome, 1920. For review see *Deut. Zuckerind.* 46, 107(1921).

Refining sugar. D. GRANT. *Brit.* 159,640, Dec. 6, 1919. Raw cryst. sugar is mixed with dry kieselguhr or decolorizing carbon, which absorbs the impurities on the outside of the crystals and yields a dry, sand-like substance. The mixt. may be sepd. by mechanical means, as, *e. g.*, by washing in a centrifugal machine with a sugar soln. to remove the absorptive substances, or the sugar may be dissolved in H_2O , which may be acidified, when the absorptive substances retain the impurities.

Starch; separating fine materials. CORN PRODUCTS REFINING CO. *Brit.* 159,838, Mar. 3, 1921. In a process and app. for sepg. gluten from starch, applicable also for sepg. other materials, the gluten and starch suspended in liquid are subjected to agitation and aeration for the production of a froth from which the starch is allowed to drain, and the froth of gluten is sepd. from the starch-contg. liquid.

29—LEATHER AND GLUE

ALLEN ROGERS

Evolution of the various methods of tanning. URBAIN J. THUAV. *Cuir* 10, 80-4, 102-6(1921); *J. Soc. Leather Trades' Chem.* 5, 70-83(1921).—A review of the development of methods of tanning by means of salts of Cr, Fe, Al, Zn, Mn, Mo, W, U, and silicic acid, pyrophosphates, aldehydes, quinones, colloidal matter in conjunction with vegetable tanning materials, and synthetic organic products of the type of Bakelite. In connection with vegetable tanning, reference is made to the Procter-Wilson theory and to slow, rapid, mixed, and vacuum tannages. J. A. WILSON

Action of sodium carbonate on chrome alum solutions. LOUIS MEUNIER. *Cuir* 10, 84-9(1921); *J. Soc. Leather Trades' Chem.* 5, 103-11(1921); *J. Am. Leather Chem. Assoc.* 16, 321-7.—The amt. of Na_2CO_3 required to start the pptn. of a freshly prepd. soln. of chrome alum decreases with diln., rise of temp., time since the Cr soln. was prepd., and vigor of agitation. The hydrolyzed Cr salts polymerize slowly on standing, but more rapidly when heated, and the liberated CO_2 is expelled more rapidly by agitation and heating. J. A. WILSON

Necessity of an international agreement on the analysis of tanning materials. MARIUS CHOPIN. *Cuir* 9, 608-10(1920).—The recent papers by Baldracco and Camilla (*C. A.* 14, 3544), by Wilson and Kern (*C. A.* 14, 2103; 15, 327), and by Schell (*C. A.* 15, 328, 608, 1088) all show that there is no sound agreement on the present official method of tannin analysis. On the same sample of ext. using the official method, the leading leather labs. of France, England, Belgium, Holland, and Switzerland obtained values for % tannin ranging from 24.9 to 28.9. This is a most serious matter in the ext. market and it is urged that an international commission be appointed to review the entire subject. J. A. WILSON

Insolubles in tanning extract analysis. L. BLEVNIÉ. *Cuir* 10, 57-8(1921).—The value for insol. matter obtained by the official method is not correct because the filter paper tends to act as an ultrafilter and retains some of the tannin. Different types of paper give very different results; for the same ext. one paper gave 2.0 and another paper 6.9% insol., with an equal difference, therefore, in the % tannin. A positive value for insol. is obtained for a liquor already filtered clear, when it is passed through a second paper. The color of the filtrate is lighter the harder the paper used and becomes still lighter with repeated filtration. It is suggested to filter the liquor once and then

filter a portion of the filtrate a second time; the drop in concn. due to the second filtration is subtracted from the drop in concn. due to the first and the difference taken as a measure of the insol. matter.

J. A. WILSON

Weak points in tannin analysis. E. SCHELL. *Cuir* 10, 107-9(1921).—Criticism made upon the method involves either the basic principle or the question of concordance. The official method gives concordant results only when the operators carry it out in a manner absolutely identical. But variations in the hide powder and the personal factor, especially in deciding when a filtrate is optically clear, lead to very large differences in the detn. of tannin. A more rigid standardization of the method is essential for concordant results.

J. A. WILSON

The time factor in the adsorption of chromic sulfate by hide substance. ARTHUR W. THOMAS AND MARGARET W. KELLY. *J. Am. Leather Chem. Assoc.* 15, 487-95(1920); cf. *C. A.* 15, 610.—A soln. of pure $\text{Cr}_2(\text{SO}_4)_3$ contg. 2.185 g. Cr_2O_3 per 100 cc. was used. When this soln. was poured on to dry hide powder as in previous work (cf. *C. A.* 14, 2563, 3336) erratic results were obtained and so the hide powder was covered with 50 cc. water and allowed to stand overnight when 150 cc. of the soln. was added making a final concn. of 1.639 g. per 100 cc. These mixts. were filtered, washed and analyzed at stated times as in previous work. The results showed an increase in Cr_2O_3 and SO_4 up to 32 days. The 64 day period showed little change. The H^+ concn. gradually decreased, due to hydrolysis since the curve is parallel to that obtained in a control liquor. Adsorption is practically complete in 32 days when 138 mg. Cr_2O_3 per g. hide substance has been adsorbed. $\text{Cr}_2(\text{SO}_4)_3$ tans much more slowly and to a lesser degree than the basic sulfate, due to the H^+ concn. which is about 20 times as great in the first case. The speed of the reaction does not conform to the simple mono-, bi- or trimolecular reaction formulas. During the 1st and 2nd hrs. the speed of reaction with SO_4 is greater than with Cr_2O_3 as in the case of a chrome liquor; after the 2nd hr. the 2 from chrome liquor are about the same while from $\text{Cr}_2(\text{SO}_4)_3$ after the 4th hr. the speed of the Cr_2O_3 exceeds that of the SO_4 .

I. D. GARARD

The determination of moisture in leather, 1920 committee report. ROBERT WRIGHT. *et al.* *J. Am. Leather Chem. Assoc.* 15, 529-34(1920).—Moisture detns. were made on sole, belting and heavily greased harness leather, prepared by cutting the leather into pieces $\frac{1}{16}$ and $\frac{1}{32}$ in. square and by planing into strips $\frac{1}{16}$ and $\frac{1}{32}$ of an in. thick. The results show that coarse particles dry properly and that moisture is liable to be lost in preparing very small pieces. Samples of a heavily greased harness leather and of a belting leather were ground on a circular saw, mixed and sent to members of the committee who dried 5 g. portions for 4, 6, 8, 10 and 16 hrs., resp. Results by Marshall showed a gradual increase in moisture with increase in time of drying. Those by Veitch and Frey dropped decidedly for the 16-hr. period, while those by Wright dropped over the 8 and 10 but gained for the 16-hr. period. This fluctuation could not be due to oxidation of oils but only to variation in moisture content caused by a change in temp. or by improper ventilation.

I. D. C.

Methods of the A.L.C.A. for the analysis of vegetable materials containing tannin. ANON. *J. Am. Leather Chem. Assoc.* 16, 113-39(1921).—The official and provisional methods of the Association are reprinted for information. They include methods of collection and prepn. of samples, and the methods of analysis for the following materials: Fresh and spent tanning materials such as wood, bark, etc., liquid and solid tanning exts., tan yard liquors, vegetable and chrome leathers, chrome tan liquors, sulfonated oils, moellons, and hard greases, and lactic acid.

J. S. ROGERS

Single-bath chrome tanning. E. GRILICHES. *Collegium* 1920, 416-29, 471-9; 1921, 127-9. See *C. A.* 14, 3819.

E. J. C.

The influence of the composition of water in its use for tanning purposes. G. GRASER. *Haute und Lederber* 1920 No. 22; *Ledertechn. Rund.* 12, 127(1920); *J. Am.*

Leather Chem. Assoc. 16, 165(1921).—Soft water according to Bitner, produces thin flat hides and hard water fixes the dirt and grease. The sulfates of Ca and Mg in general produce good swelling, while their carbonates are detrimental. Water containing much carbonate is best neutralized with H_2SO_4 , but $CaSO_4$ yields a leather with a coarse feel and one that is difficult to dye. The question of the effect of hard water on the extn. of tanning materials is still open. Chlorides arrest the swelling of hide, make tanning difficult, and when present in large quantities attract moisture. Water containing bacteria which attack protein, putrefactive bacteria which attack corium and liquefy hide substance is dangerous to use in the tannery. Water may be treated by boiling, by chemical softening, or sterilizing by ozone. J. S. ROGERS

Tannage of rabbit skins and fur. GABRIEL DESMURS. *J. Soc. Leather Trades' Chem.* 5, 84-7(1921).—The skins of rabbits and similar animals are tanned in liquors made up from: quebracho ext. (unsulfited-40% tannin) 100 parts; sumach ext. (22% tannin) 10 parts; Na_2SO_4 (anhydrous) $2\frac{1}{2}$ parts; Al_2SO_4 $\frac{1}{2}$ part. A similar mixt. is suitable for tanning sheep skins. There is also given a short account of fur dyeing methods. JEROME ALEXANDER

Clarifying and bleaching glue. ANON. *Farben-Ztg.* 26, 1459(1921).—If hot glue solns. do not become clear on standing, impurities may be pptd. by addition of alum; this has the further advantage of preventing decompn. on slow drying of the glue. Oxalic acid, ext. of hops or oak bark may also be used but these ppt. a part of the glutin and, therefore, weaken the glue. Decolorization by animal charcoal is only used in special cases and then a preservative must be added to prevent decomp. during filtration. Treating the crude stock with Cl or SO_2 is a more satisfactory method for obtaining a light colored product but this produces a glue of acid reaction, which is not desirable for certain uses. F. A. WERTZ

Analysis of sulfonated oils (BUMCKE) 27. Improved formulas for the calculation of inorganic impurities in sulfonated oils (HART) 27. Preservation of fish nets (TAYLOR) 25.

GRASSER, GEORG: *Synthetische Gerbstoffe, ihre Synthese, industrielle Darstellung und Verwendung* Berlin: Verlag von Hermann Meusser. 154 pp. M. 84.

THEVAU, URBAIN J.: *L'évolution des différentes méthodes de tannage.* Paris: Le Cuir, 54 rue de Bondy. 20 pp. For review see *Rev. prod. chim.* 24, 272(1921).

Leather substitute. EWALD ARNOLD. *Ger.* 322,987, Dec. 21, 1917. Layers of paper are satd. with a mixt. of animal glue with over 50% of sulfite waste lye, to which HCHO is added. The HCHO may be employed in the usual commercial concn. or in a 10% diln. The sulfite waste liquor affects the suppleness of the final product very favorably.

Tanning skins and hides. SOCIÉTÉ GENTY, HOUGH & Co. *Ger.* 322,166, Aug. 17, 1918. The skins or hides are satd. with a silicate soln. containing 2.5-10% of Na or K silicate of the usual commercial concn. When the skins have been left in the water-glass soln. for several hrs. they swell considerably. The pptn. of silicic acid should be effected slowly and by means of substances which will not harmfully affect the skins, like most of the mineral or org. acids. HOAc acid has proved to be most suitable for the purpose. The tanning process is completed when the white color extends uniformly over the entire cross-sectional surface of the skin, hence from the flesh side to the grain side. When the tanning operation has been finished, the skins are rinsed off, allowed to drip, and impregnated with a paste the compn. of which depends on the degree of suppleness to be imparted to the leather.

Quick tanning of skins. FELICE GILARDINI. *Norw.* 30,604, Apr. 26, 1920. On one

side of the skin a tanning soln. is made to act under pressure, while the other side is kept under atm. pressure or a vacuum. The surface of the skin is subdivided by suitable app. into a plurality of zones which are tanned independently of one another. Means are provided for isolating the places where the skin shows defects or imperfections in order to prevent quick tanning from taking place around such points.

Tanning material. VIGGO DREWSSEN. Norw. 30,921, Apr. 26, 1920. Lignin or lignin-containing substances produced from wood are dissolved in a monosulfitic solvent and the tanning matter is pptd. by addition of acid.

Tanning material. CARL GRAF. Ger. 322,387, Mar. 4, 1916. Vegetable tar is lixiviated with an aq. soln. of sulfites more particularly with Na_2SO_3 . The substances which are of value in tanning dissolve therein, while the phenols of the tar are not dissolved. The tanning liquor is further improved by addition of alum or similarly acting acid salts, such as $\text{Al}_2(\text{SO}_4)_3$, chrome alum, or the like, and CuSO_4 . By raising the temp. the process is accelerated.

Tanning with iron salts. OTTO RÖHM. Norw. 30,740, May 18, 1920. In combining the process of iron salt and aldehyde tanning with a sulfide or polysulfide treatment of the skins according to Norwegian patent 29,849, there is employed, in addition to the treatment of the skins with sulfides or polysulfides, an oxidation process which consists, either in laying the damp leather in the open air or in treating the same with other oxidizing agents.

30—RUBBER AND ALLIED SUBSTANCES

JOHN B. TUTTLE :

Apparatus for determining the permeability of gas-tight materials (FRENZEL) 1. Drying colloids (Brit. pat. 159,465) 13.

BÉGIN, M. J.: *Analyse des caoutchoucs bruts et manufacturés*. Paris: Ch. Beranger. 47 pp. 5 fr. For review see *Bull. soc. ind. Rouen* 48, 635(1920).

SMITHSON, H. E.: *The Cultivation, Chemistry, Testing and Manufacturing of Rubber, with Sections on Reclamation of Rubber and the Manufacture of Rubber Substitutes*. New York: D. Van Nostrand Co. 149 pp. \$4.50.

Rubber Goods Manufacture. By "FACTORY MANAGER." London: MacLaren & Sons. 496 pp. For review see *Rubber Age* 9, 90(1921).

Preparation of rubber. S. C. DAVIDSON. Brit. 159,602, Dec. 1, 1919. Latex, as soon as practicable after being tapped, is treated with an alk. phenol soln. as described in 11,470, 1912 (C. A. 7, 3673) and 13,438, 1913 (C. A. 8, 3867) and is subsequently coagulated by the addition of an acid or acid salt, e. g., an acidified soln. of a Mg salt, preferably the sulfate. This treatment may be carried out in the app. described in 22,489, 1914 (C. A. 10, 1289). The coagulum is dehydrated mechanically preferably in app. described in 151,344 (C. A. 15, 1831) and 159,106 in which a layer of rubber is perforated by pins carried by a plate which is operated to press the rubber and express liquid entrapped in the rubber through the perforations thus produced. The dehydrated rubber may be dipped into hot H_2O to soften it and is then sheeted or creped, and may be marketed in this form or rolled up and kneaded into cylinders, blocks, etc., preferably by the app. described in 16,400, 1915. The whole process may be carried out on the estate, or the preserved latex may be exported and the remainder of the process carried out elsewhere.

Reclaiming rubber. HERMANN PENTHER. Norw. 31,274, Oct. 18, 1920. The rubber is rolled out to the thickness of a film at least 20 times, so that it is completely

opened out and may be used anew; and also vulcanized, either by itself or in combination with new rubber or fillers.

Vulcanizing rubber. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. Ger. 306,667, Feb. 20, 1917. To the masses to be vulcanized are added sulfates of aniline or of its homologs and derivs., in certain cases also amines or NH_4 compds. of aliphatic or aromatic nature. Example: 100 parts by wt. of isoprene rubber are heated with 5 parts by wt. of S and 1 part by wt. of aniline sulfate for 60 mins. to 3 atm. or, 100 parts by wt. of regenerate are heated with 5 parts by wt. of S and 1 part by wt. of aniline sulfate for 30 mins. to 3 atm. In place of aniline sulfate, toluidine sulfate may be employed, and the isoprene rubber may be replaced by rubber obtained from erythrene or dimethylethyrene or crude rubber.

Vulcanizing rubber, regenerate or the like on metals. FELTEN & GUILLAUME, CARLSWERK, AKT.-GES. Ger. 326,541, Oct. 10, 1918. An intermediate layer consisting of a sticky viscous balsam-like mass which is not absorbed by rubber is interposed between the metal and the rubber.

Producing soft, elastic and stretchable vulcanisates. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. Ger. 303,984, Feb. 20, 1917. To the masses to be vulcanized are added NH_4 compds. or amines of aliphatic or aromatic nature, or their derivs. and salts in amts. of about 5% and above. Example: 100 parts by wt. of isoprene rubber are heated with 5 parts by wt. of S and 10 parts by wt. of Ph_2NH for 30 min. to 3 atm., or 100 parts by wt. of regenerate are heated with 5 parts by wt. of S and 10 parts by wt. of PhNEt_3 for 30 min. to 3 atm.

